

Exhibit 137

TRAVELING EXPENSE REPORT

INSTRUCTIONS:

1. Print in Ink or Type. No Alterations or Erasures are permitted.
2. Report only Expenses actually incurred on Company business. Enter each day's expenses separately.
3. Attach All Receipted Bills. Give Names of recipients of substantial amounts not covered by receipts.
4. Submit Report Weekly. If trip is less than one week submit Report promptly upon completion of Trip.
5. Obtain Written Approval on this form prior to travel.

NAME <i>Ian M. Stewart</i>	CHARGE TO <i>MA2546</i>	PERIOD ENDING <i>Sept. 21, 1972</i>
PURPOSE OF TRIP <i>Washington meeting - J & J - F. D. A.</i>		

1. Date	<i>20th Sept</i>	<i>21st Sept.</i>																		
2. Cities visited	From	<i>Chicago</i>	<i>Washington</i>																	
	To	<i>Washington</i>	<i>Chicago</i>																	
3. Auto Mileage—Actual mileage for business purposes.	<i>20</i>	<i>23</i>																	TOTAL	
4. Auto Mileage Expense—See note #1 below.	<i>2</i>	<i>60</i>	<i>2</i>	<i>99</i>															<i>5</i> <i>59</i>	
5. Train, Bus, Plane fare, or Auto Rental—Only if paid by traveler. See note #2 below.																				
6. Hotel or Lodging—Enter each day's expenses separately. Show hotel room and meals separately.																				
7. Meals—Personal meals only. Include tips.																				
8. Telephone or Telegraph—For telegraph or long distance telephone, show name of person contacted on reverse of form.																				
9. Taxi or Bus fare			<i>4</i>	<i>00</i>															<i>4</i> <i>00</i>	
10. Guests—Show names of guests and full details on reverse of form.																			<i>5</i> <i>40</i>	
11. Other Expenses—See note #1 below. Show expenses separately on reverse of form.																				
12. Total Expense Paid by Traveler																			<i>14</i> <i>99</i>	
13. Transportation paid by Company—Do not include Item 5 above.																			<i>132</i> <i>00</i>	
14. Auto Rental paid by Company—Do not include item 5 above. (Show ticket number or invoice number if required internally)																				
15. Are unused Transportation Tickets attached? <input type="checkbox"/> Yes <input checked="" type="checkbox"/> No If Yes, show estimated value of tickets and deduct to get Total Expense (Item S1). Unused Tickets must be attached to this Report and a full explanation given on reverse of Form. They will be forwarded by Accounting for Claim.																				
OK for Payment	Initial																S1. Total Expense (Lines 12, 13 and 14, Less 15)		<i>146</i> <i>99</i>	
																	S2. Cash Advanced By Company		<i>50</i> <i>00</i>	
																	S3. Transportation Paid By Company (Lines 13 and 14, Less 15)		<i>132</i> <i>00</i>	
																	S4. Total Advanced By Company (S2 + S3)		<i>182</i> <i>00</i>	
																	S5. Balance Due Traveler (S1 in excess of S4)		<i>—</i>	
																	S6. Balance Returned to Company (S4 in excess of S1)		<i>35</i> <i>01</i>	

NOTES

1. Show Parking, Tolls and other allowable items separately on Line 11. Do not include Gasoline, Oil, Repairs, etc.
2. If Paid by Company, enter on Line 13.

I request to be reimbursed for expenses incurred by me on behalf of McCrone Associates in the sum of (Line S5):

AMOUNT MUST BE WRITTEN OUT

SIGNATURE OF TRAVELER (in ink)

Ian M. Stewart

DATE SIGNED

Sept. 28, 1972

AUTHORIZED APPROVER

DATE OF APPROVAL OF TRAVEL

Exhibit 138

CONFIDENTIAL

MARCH, 1974

To: Windsor Minerals Inc., Windsor, Vermont 05089

From: R.C. Reynolds Jr., Department of Earth Sciences,
Dartmouth College, Hanover, New Hampshire 03755

Subject: Analysis of Talc Products and Ores for Asbestiform
Amphiboles

INTRODUCTION:

The purpose of this study is to develop methods for measuring the concentration of asbestiform amphiboles in fine-grained talc products and talc ores. In principle, the problem might be simply solved by the microscopic counting of amphibole grains in samples that are suspended in oils of suitable refractive index, so that amphiboles are optically emphasized with respect to talc. In practice, however, grain counting is valid only if the dimensions of each grain are measured. This requirement arises from the large particle size range present, and from the wide variation of aspect ratios among the amphibole grains (see plate 16). In any event, grain-counting methods are inapplicable to whole samples because an inordinate number of grains must be considered. This makes the analysis time prohibitively long. For example, if a concentration of 100 ppm amphibole is assumed, and if 100 amphibole grains is the acceptable minimum that provides good statistical data, then one million grains must be considered. This requirement would be

easily met if the amphiboles were easily distinguished from other minerals. But even in suitable refractive index oils, many grains are ambiguous and require manipulations in order to verify their identity. Such ambiguous grains are (1) fibrous talc and carbonate grains in certain orientations (plate 21), (2) inclusion-filled grains (plate 21), (3) broken amphibole grains that have equant shapes (plate 9), and (4) grain aggregates (plate 20). If only 1% of the grains are ambiguous, and experience indicates that this is a conservative figure, then the example cited above would demand that attention be paid to 10,000 grains in a single sample.

For the reasons described above, a concentration technique is mandatory because it brings the amphiboles into a reasonable concentration range for optical or other methods of analysis. Such a method has been developed, and it is described in this report.

EXPERIMENTAL METHODS:

Advantage can be taken of difference in density between talc and amphiboles (see Table 1). The sample can be suspended in bromoform and centrifuged to float talc and settle denser minerals. However, the fine-grained nature of the ground ores and products brings colloidal forces into play which cause flocculation of the sample, and this renders a clean separation impossible.

Dispersion of talc in bromoform requires that the particles

be plated with organic molecules whose film thicknesses separate the grains sufficiently so that van der Waals forces cannot cause agglomeration. In addition, the plated grains must make up an oleophilic colloid, that is, they must be wettable by non-polar organic solvents (bromoform). Experiments were performed with various concentrations of butylamine hydrochloride, cetyltrimethylammonium bromide and benzethonium chloride monohydrate. The latter reagent was far superior in promoting dispersion in bromoform. The effectiveness of dispersion was judged by the time required for visual evidence of flocculation of the talc product.

The addition of benzethonium chloride monohydrate (hereafter, abbreviated BCM) lowered the density of the bromoform. Methylene iodide was added to bring the density back to desired levels. The proportions of sample and reagents used for the separations described in this report, are:

1 g talc,
2.0 g BCM,
20 ml Bromoform ($d=2.8$) and
8.4 ml Methylene iodide ($d=3.3$).

This mixture provides a density of 2.88 g/cm^3 , at 20°C , as measured by pycnometer. Attempts to raise the density by further additions of methylene iodide promoted flocculation. Consequently, future separations of amphiboles should be made at reagent concentrations similar to these.

Separations of amphiboles were made from ground talc product and from talc ore, provided by V. Zeitz of Windsor Minerals.

Talc was weighed into centrifuge tubes, and the reagents added in the proportions cited above. The tubes were shaken vigorously for approximately one minute, placed in a size 2 International Centrifuge, and centrifuged for 5 minutes at 500 RPM, followed by 5 minutes at 1800-2000 RPM. The centrifuge was allowed to slow with no braking (to minimize counter-rotating currents in the liquid) and the tubes were withdrawn and placed in racks for isolation of the heavy mineral fractions.

A glass rod was fitted with a rubber stopper, and this was carefully inserted into the tube and lowered to the bottom so as to isolate the heavy fraction (See Figure 1). The tube was decanted* and flushed with acetone from a polyethylene wash bottle. The plunger was removed, washed with acetone into the tube, and the tube was filled with acetone and shaken and centrifuged. The heavy fraction was washed twice more with acetone by means of the centrifuge, decanted, and dried overnight at 80°C. The tube with sample was cooled in a dessicator and weighed on an analytical balance. The concentrate at this stage consisted mostly of carbonate (magnesite plus some dolomite, see Plate 5). The carbonates were removed by acid dissolution as described below.

The sample tube was filled with 4NHCl and heated in a water bath for 2 hours at 80-90°C. The sample was centrifuged

* The reagent mixture cannot be saved. Slow decomposition occurs which liberates iodine and reduces the density. After 24 hours this process is sufficiently severe to lower the density below that of talc.

twice more to wash soluble salts out of the insoluble heavy mineral fraction. It was dried overnight at 80°C. The sample was scraped out of the tube, transferred to a vial, and the centrifuge tube was washed, dried, and weighed. From the three weights obtained, values were calculated for (1) percent heavy minerals, and (2) percent insoluble heavy minerals.

Talc ore and talc product, provided by V. Zeitz of Windsor Minerals, were run through this procedure. In addition, talc ore was spiked with known amounts of actinolite (ground and sized 2-2 μ by settling in water), and separated to test the efficiency of the method. The concentration of actinolite in the product concentrate was estimated by optical examination of the insoluble heavy mineral fraction. A simple estimate was made, and this was normalized to the total sample weight by means of the figure for the percentage of the total represented by the separate. Values for actinolite in the ore and in the spiked samples of ore were obtained by an X-ray fluorescence method that utilizes known amounts of potassium added as an internal standard. X-ray fluorescence methods were used because of the failure of various X-ray diffraction methods which were attempted. All of these had unacceptable precision ($\pm 100\%$), probably due in most part to the small (milligram) amounts of material available for analysis. Consequently, the X-ray fluorescence method provides the best means of measuring actinolite, although it would be useless for the determination of other fiberform amphiboles.

RESULTS:

Two-gram samples of ore and ore spiked with actinolite were separated and analyzed as described above. Results for these samples are summarized by Table 2. Table 3 shows concentrations of heavy fractions (mostly carbonate), and of actinolite in talc ore and talc product. Actinolite concentration in the product was measured for a 12 gram sample.

DISCUSSION:

The data of Table 2 show that (1) the total amount of heavy minerals separable from talc ore is reproduceable, and (2) the amounts of actinolite recovered from spiked samples agree quite closely with the actual amounts added. The agreement between actinolite found and actinolite nominal is about as good as can be expected, given the existence of errors of one or more milligrams that can arise as a result of the weighing procedures described above.

The samples studied contain actinolite as the dominant fiberform amphibole phase. This conclusion is based upon (1) the common occurrence of extinction angles of 15° , (2) the α refractive index of 1.616, and (3) the high calcium content of the residues which contained no other calcium-bearing minerals. However, some anthophyllite might be present in very small amounts (See Plates 7 and 8). It was identified on the basis of (1) parallel extinction, (2) extreme aspect ratio, and (3) lack of the length-parallel striations that characterize

actinolite.

The data of Table 1 show that cummingtonite and anthophyllite can have densities as low as 2.85, and these would float with talc in the liquids used (density = 2.88). But these low densities are for amphiboles that are pure magnesium end-members, and as such, are mineralogical curiosities that are very rare in nature. It is likely that the usual compositions of anthophyllite and cummingtonite would be closer in density to actinolite. Because all amphiboles have similar surface chemistry, the separation techniques described here would almost certainly work for their concentration from talc and talc ore matrices. In fact some progress has been made to that end, though the results are preliminary and can only be described as promising.

CONCLUSIONS:

1. Mixtures of bromoform, methylene iodide, and benzethonium chloride monohydrate provide a suitable heavy liquid for the centrifugal separation of fiberform amphiboles from talc in samples composed of clay to silt-sized grains.
2. The ore sample contains 2300 ppm actinolite, and the talc product contains ~170 ppm actinolite.
3. Actinolite is the dominant fiberform amphibole

in the ore and talc product provided by Windsor Minerals. Small amounts of anthophyllite may be present.

4. Calcium analyses on acid-treated heavy mineral residues serve as an effective means of determining actinolite.
5. The determination of crocidolite, cummingtonite, or anthophyllite in concentrates is probably best accomplished by a microscope method.

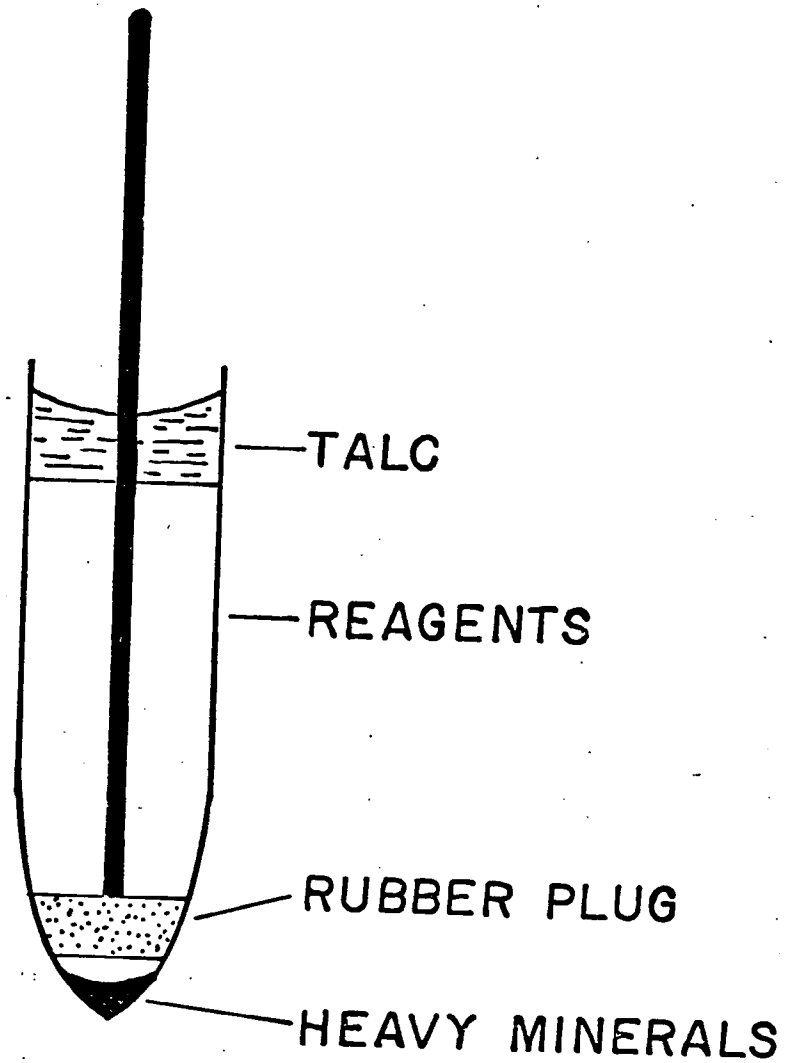


Figure 1. Apparatus for isolation of heavy minerals from centrifuge tube.

TABLE 1 - Densities of Common Fiberform Amphiboles
and Talc (Hurlbut, C.S., 1971, Dana's Manual of
Mineralogy, 18th Ed., Wiley).

Mineral	Density
Talc	2.7-2.8
Tremolite-Actinolite	3.0-3.3
Cummingtonite	2.85-3.2
Anthophyllite	2.85-3.2
Crocidolite	3.2-3.3

TABLE 2 - Residue Weights and Actinolite in Talc
Ore and Actinolite-Spiked Talc Ore.

Sample	Heavy Minerals; mg/2g Sample	Acid Insoluble Heavy Minerals; mg/2g Sample	mg Actinolite found*	mg Actinolite nominal
Ore	478	14.3	4.6	---
Ore + 4.3 mg Actinolite	445	19.8	7.8	8.9
Ore + 8.6 mg Actinolite	458	20.4	9.8	13.2
Ore + 12.9 mg Actinolite	459	30.1	17.3	17.5

*Based on a nominal value of 9.7% Ca in actinolite,

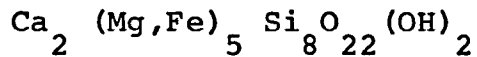


TABLE 3 - Heavy Fraction and Actinolite in Talc.

Sample	% Heavy Minerals (~Carbonate)	PPM Actinolite
Talc Ore	23.9	2300
Talc Product	--	170

APPENDIX

Photomicrographs have been taken of the minerals in talc and talc ores. Attempts have been made to catalog the dominant mineral species, and to depict the range in form characteristic of each mineral. For both talc product and talc ore, photos have been taken of (1) the bulk materials, (2) the heavy mineral fractions, and (3) the acid insoluble heavy mineral fractions. The photos below are accompanied by brief descriptions of each, and some mineral grains are appropriately labelled according to the following key.

Talc	T
Carbonate	C
Chromite	Cr
Actinolite	Ac
Anthophyllite	An
Epidote	Ep

Although the forms of the minerals shown on the photos are characteristic, many of the photos show non-representative mineralogical compositions. For example, a good deal of searching was required to find actinolite and carbonate grains in bulk talc products (Plates 14 and 11). Similarly, anthophyllite is extremely uncommon and considerable amounts of sample must be examined in order to find one grain (Plates 7 and 8).

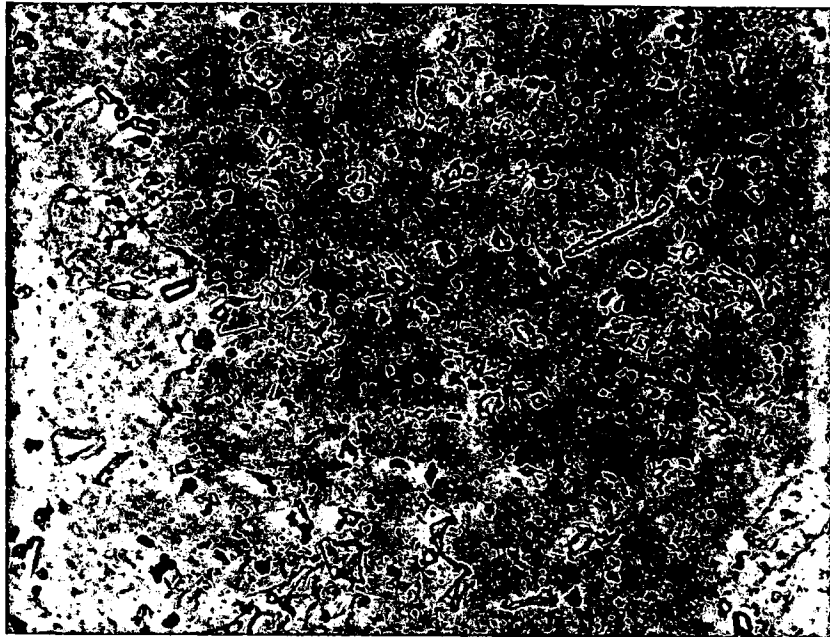


Plate 1

Talc Ore, Bulk; x 100; $n=1.503$
Low relief background is talc, high relief equant
grains are carbonate, and the long grain in the
upper right corner is actinolite.



Plate 2

Talc Ore, Bulk; x 400; $n = 1.503$
Platy talc, actinolite, and carbonate.
Note the length-striated character of actinolite;
this is characteristic.

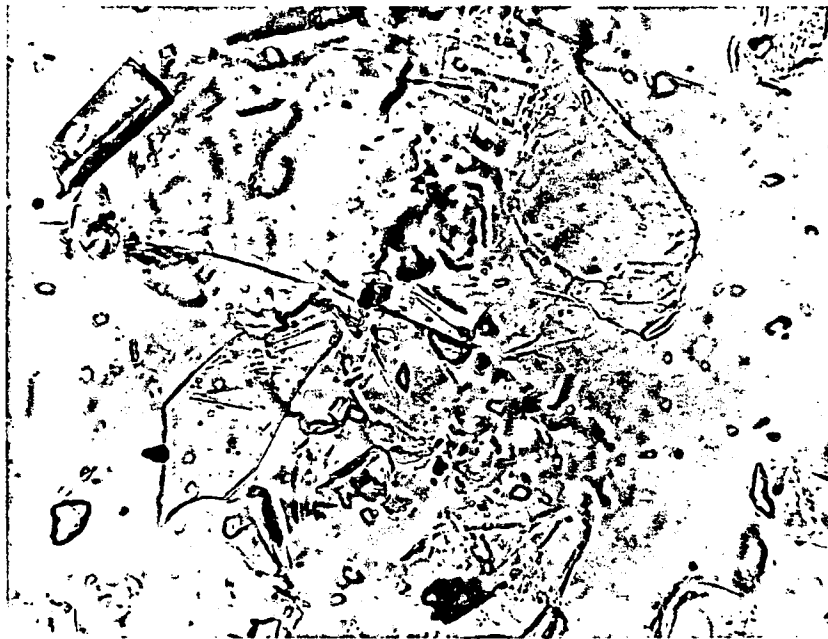


Plate 3

Talc Ore, Bulk; x 400; $n=1.503$
Platy talc and carbonate. The talc
is inclusion-free and shows a well-
developed platy morphology.



Plate 4

Talc Ore, Heavy Fraction; x 400; $n = 1.503$
The field shows epidote, carbonate, talc,
chromite, and actinolite.

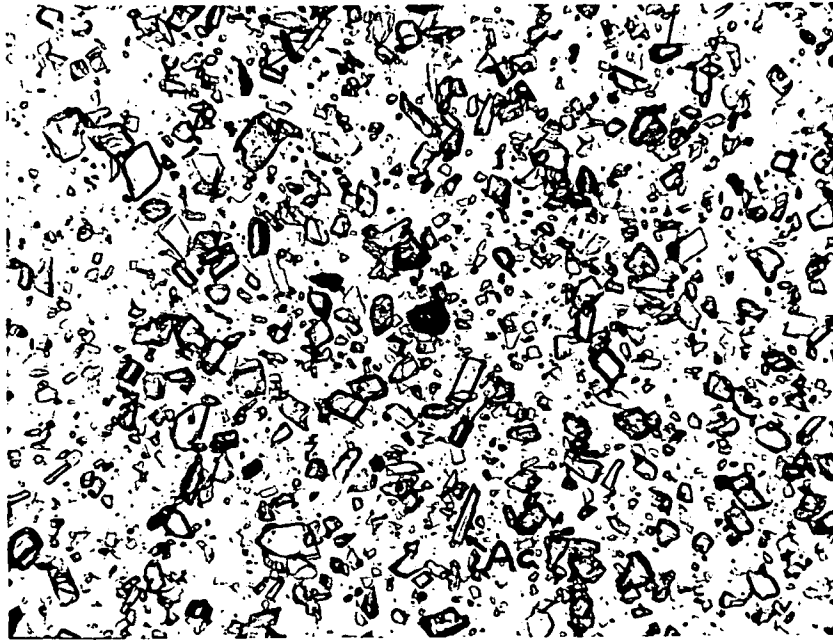


Plate 5

Talc Ore, Heavy Fraction; x 100; $n = 1.503$
This photo shows the general character of the typical heavy mineral fraction. Small amounts of talc are invisible in the background. Carbonate is the dominant mineral. Opaque grains are chromite, and actinolite is labelled Ac.



Plate 6

Talc Ore, Heavy Fraction; x 400; $n = 1.503$
This view shows mostly carbonate, some chromite
(opaque) and an actinolite grain of typical mor-
phology at the center.



Plate 7

Talc Ore, Acid Insoluble Heavy Fraction; x 100; $n = 1.503$
Actinolite, talc, chromite, and a large anthophyllite fiber.
Note that much of the talc is of poor morphology and/or
is inclusion-filled.



Plate 8

Talc Ore, Acid Insoluble Heavy Fraction; x 400; $n = 1.503$
Inclusion-filled talc, actinolite and anthophyllite.



Plate 9

Talc Ore, Acid Insoluble Heavy Fraction; x 400; $n = 1.503$
Platy talc and actinolite. Note that the small equant
grains of actinolite could be easily mistaken for carbonate.

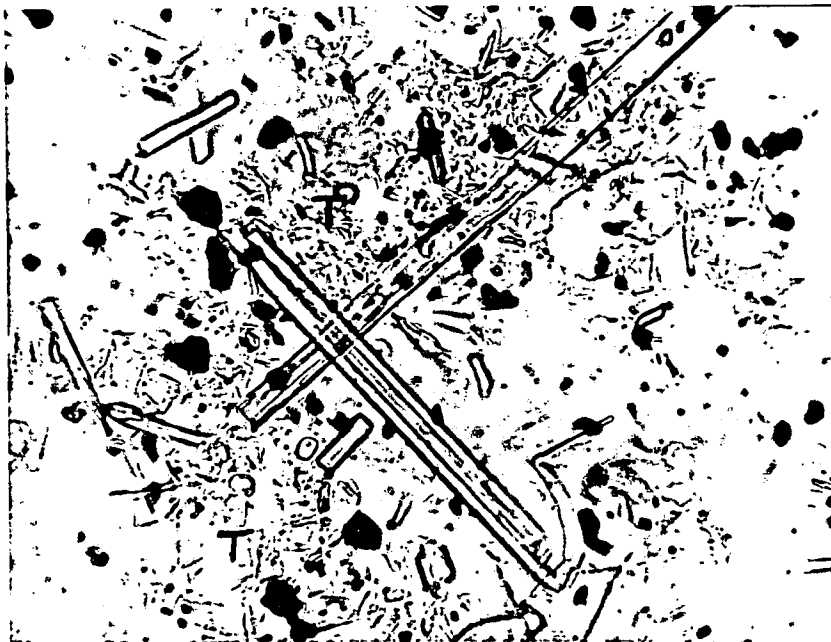


Plate 10

Talc Ore, Acid Insoluble Heavy Fraction; x 400; $n = 1.503$
Inclusion-filled talc with partial fibrous morphology,
platy talc, and characteristic grains of actinolite.



Plate 11

Talc Product, Bulk; x 100; $n = 1.503$
Platy talc with a few carbonate grains
(high relief).



Plate 12

Talc Product, Bulk; x 400; $n = 1.503$
Typical platy talc of good morphology.



Plate 13

Talc Product, Bulk; x 400; $n = 1.503$
Platy talc and carbonate.



Plate 14

Talc Product, Bulk; x 400; n = 1.503
Platy talc and one actinolite grain.

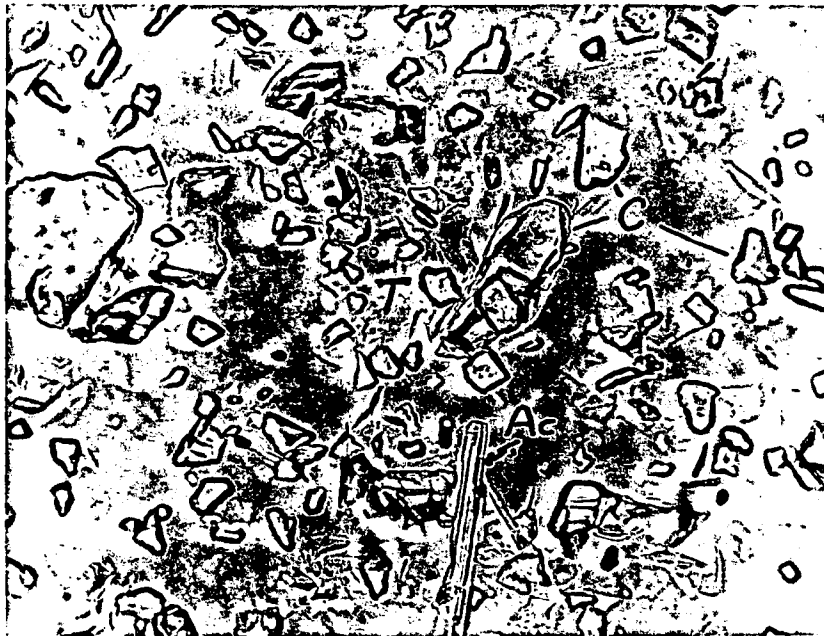


Plate 15

Talc Product, Heavy Fraction; x 400; $n = 1.503$
Typical field showing carbonate, talc, actinolite,
and chromite.

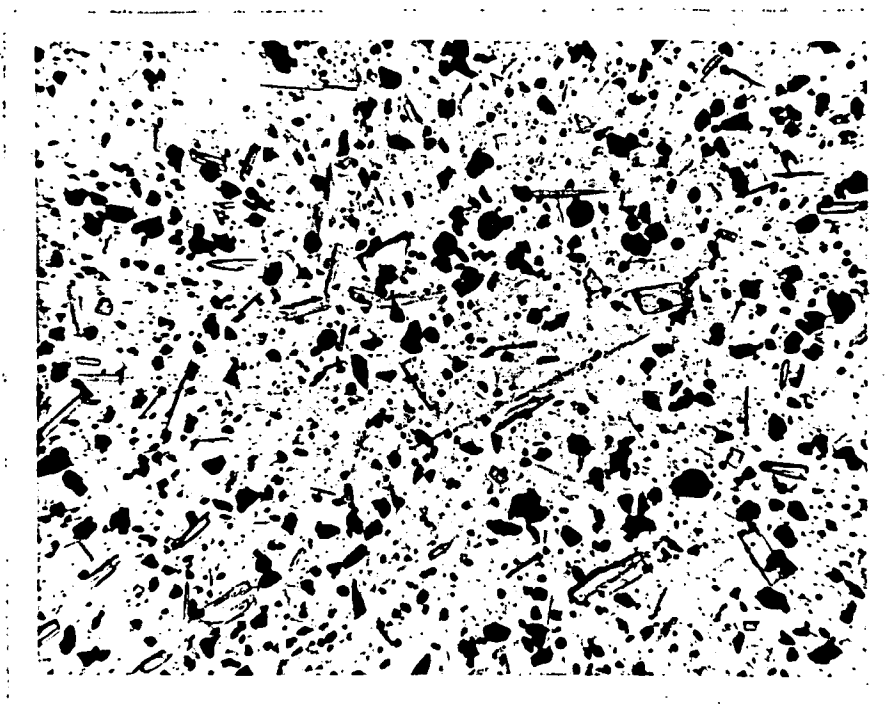


Plate 16

Talc Product, Acid Insoluble Heavy Fraction;
x 100; $n = 1.571$
Talc is invisible because of high index oil used.
Visible grains consist of chromite (opaque) and
actinolite. Note the large variation in aspect
ratio of the actinolite.

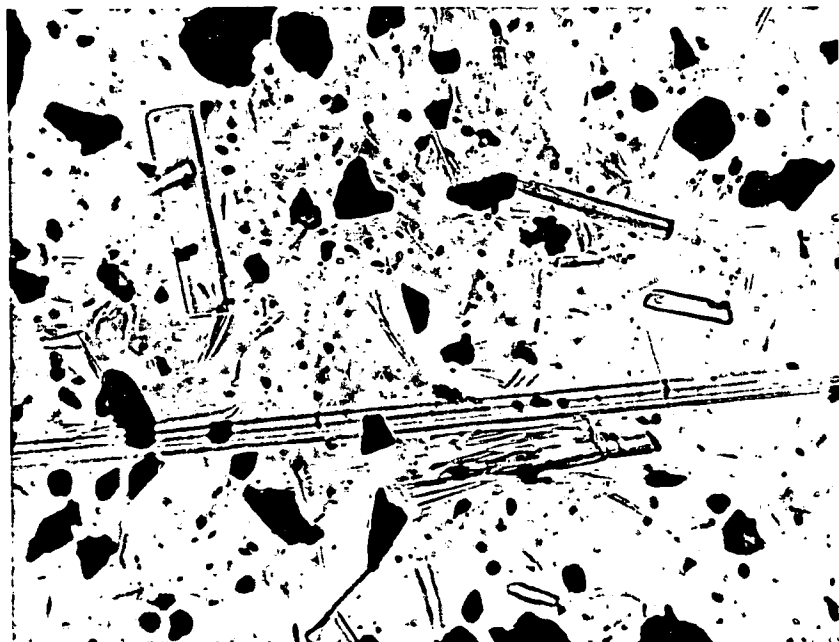


Plate 17

Talc Product, Acid Insoluble Heavy Fraction;
x 400; $n = 1.571$
Chromite and actinolite of varying morphology.
The characteristic striations are clearly visible
in the actinolite grains.

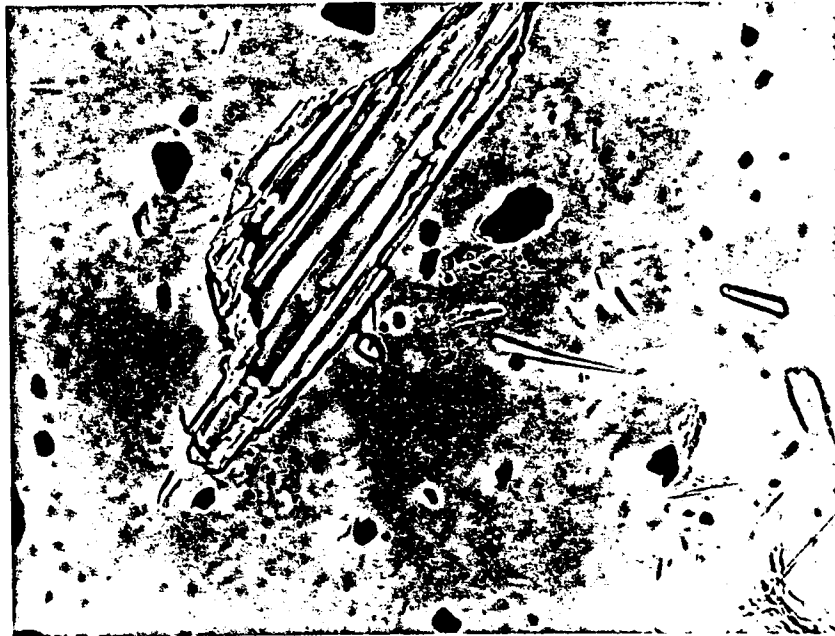


Plate 18

Talc Product, Acid Insoluble Heavy Fraction;
x 400; $n = 1.571$
Large actinolite grain with irregular shape.

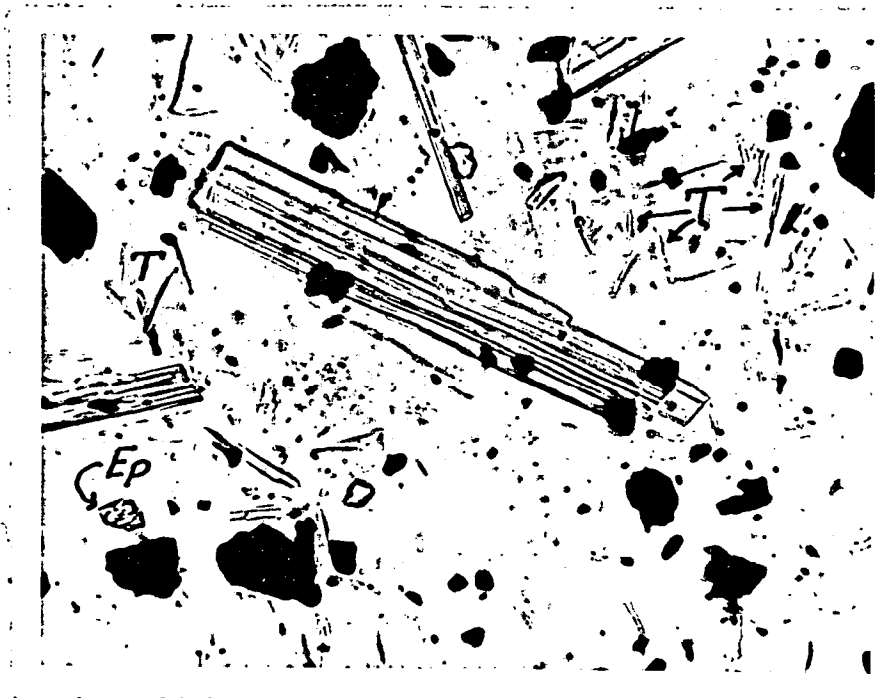


Plate 19

Talc Product, Acid Insoluble Heavy Fraction;
x 400; $n = 1.571$
Typical actinolite, fibrous talc, chromite and
epidote.



Plate 20

Talc Product, Acid Insoluble Heavy Fraction;
x 400; $n = 1.503$
Note the large compound grain (platy talc and
actinolite) at bottom center. Other minerals
are platy talc (very low relief) actinolite,
and chromite.



Plate 21

Talc Product, Acid Insoluble Heavy Fraction;
x 400; $n = 1.503$

Small talc fibers, platy talc, and inclusion-filled talc plus chromite and actinolite. It is the presence of grains such as the inclusion-filled talc that makes grain counting analysis difficult.

Exhibit 139



ASBESTIFORM AMPHIBOLE MINERALS IN COSMETIC TALC

Part I: X-ray Diffraction Method

Part II: Optical Microscopy and Dispersion-Staining Method

Introduction

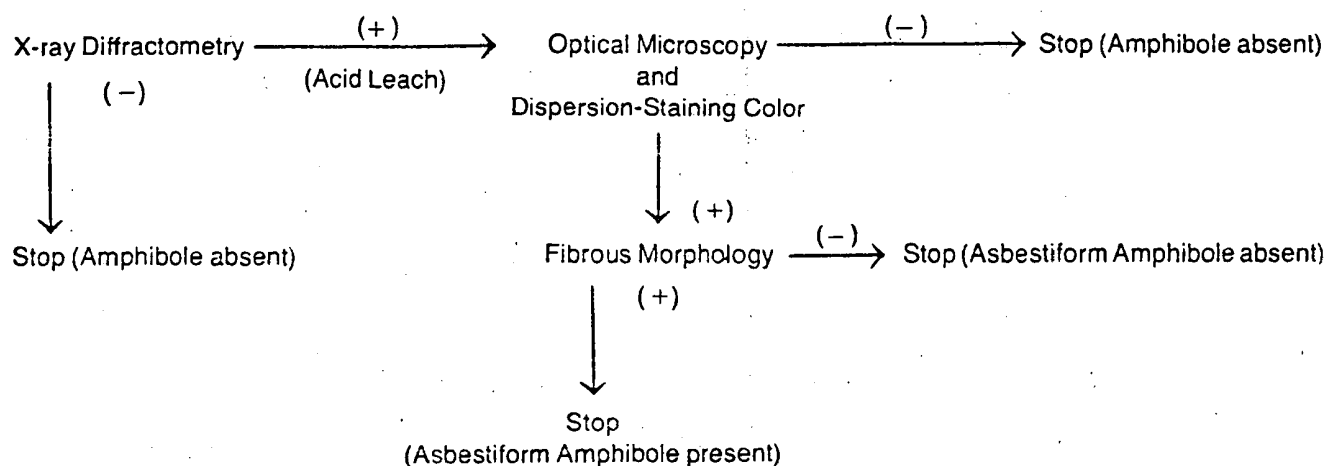
The method which has been adopted for the detection of amphibole minerals in cosmetic talc is the generally accepted method of x-ray diffraction. Methods which appear in the literature for the detection of fibrous amphibole, such as, transmission electron microscopy with selected area diffraction¹ and electron microprobe,² have also been considered since they are capable of a lower level of detection than by x-ray diffraction. However, they have not been adopted since they suffer from the drawbacks, that the amount of material under examination is quite small (less than a microgram) and the time for analysis, expertise required, and expense of equipment eliminates them as routine methods.

The methodology presented is the most practical available, based on current technology. The use of Transmission Electron Microscopy with Selected Area Electron Diffraction offers greater sensitivity, but is not presented since it is unsuitable for normal quality control application.

Enrichment or concentration techniques using flotation cells have been tried as a means of improving the detection level; however, all efforts so far have been unsuccessful.

Principle

The x-ray diffraction method is based upon the principle that when a crystalline material is placed in an x-ray beam, a portion of the x-rays are diffracted by each set of atomic planes within the crystal. The diffracted rays strike a scintillation counter as the sample is scanned through a prescribed angle with the resulting development of peaks corresponding to each interplanar distance (d). A peak with d value in the range of 8.04 to 8.85 Å for a sample talc is strong evidence for the presence of amphibole in that talc. The level of detection of amphibole by this method is 0.5% and above. The variability of detection is caused by such factors as age and manufacturer of x-ray diffractometers, sample homogeneity, specific amphibole mineral present, morphology of amphibole, particle size, preferred orientation, etc. For these reasons the level of detection should be reported for levels above 0.5%, since below this level the data has been found to be not reproducible. If a statistically significant peak is found of intensity equal to or greater than that obtained for the 0.5% standard in the d range for amphibole, described above, then the sample must be put through the following confirming scheme:



Part I: Amphibole Minerals by X-ray Diffractometry

Apparatus

1. X-ray diffractometer, employing nickel-filtered copper K-alpha radiation, horizontal or vertical goniometer with variable scan speed capability, suitable talc pellet sample holder, variable speed recorder, electronic panel including ratemeter and variable attenuation and time constant settings
2. Hydraulic press, capable of attaining a pressure of 15,000 to 24,000 lb calculated on a 3" ram
3. Mortar and pestle or grinding mill (Note 1)
4. Waring Blendor,* or equivalent blender
5. Spex Mixer/Mill,* or equivalent mechanical mixer
6. Sieve, 325-mesh
7. Optical microscope (Note 2)
8. 1 1/4" pellet press

Reagents

1. Standard talc sample, containing no detectable amphibole minerals
2. Standard tremolite sample, at least 80% pure
Sample of tremolite standard may be obtained by ordering from The Cosmetic Toiletry and Fragrance Association, Inc., 1133 Fifteenth Street, N.W., Washington, D.C. 20005.
3. Denatured ethanol
4. Boric acid

Procedure

The procedure consists of slow-scanning, under previously determined conditions, a compressed pellet of the sample talc in the 11.0 to $10.0^\circ 2\theta$ (8.85 to 8.04\AA) region for the presence of an amphibole peak. There are times when it is difficult to discriminate a possible peak for amphibole over the background noise level.

*Registered Trademark

Should the presence of a small amphibole peak above the background "noise" be in question, it will be necessary to statistically evaluate the scan. A timer/scaler is required on the electronic panel of the x-ray diffractometer. In order for a peak to be statistically significant, the peak intensity must equal or exceed three standard deviations (3σ) above the average background intensity (N):

$$N + 3\sigma = \text{minimum peak intensity}$$
$$N = \text{average background count}$$

Where:

$$\sigma = \sqrt{N}$$

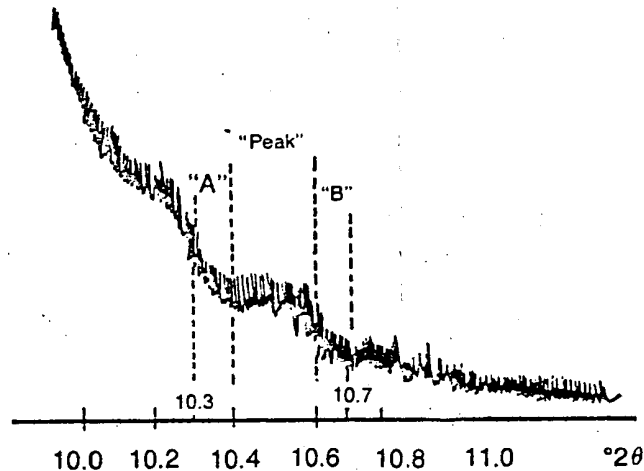


Figure 1.

Determine the region of the scan in question: in the Figure 1 scan, a peak appears to be present in the 10.40 to 10.60 2θ region.

Slow scan with cumulative pulse counting through the peak region three separate times and average the number of counts.

Determine a background count by scanning a region equal to $\frac{1}{2}$ of the 2θ region covered by the peak, immediately before and after the peak. The counting time for each of these background regions will equal $\frac{1}{2}$ the total counting time used for the peak. Count each background region three times. Then average each region and add the two averages to obtain the background count (N).

Example:

In Figure 1.

	Region ($^{\circ}2\theta$)	Time (sec.)
Peak	10.40 to 10.60	120
Background		
Region A	10.30 to 10.40	60
Region B	10.60 to 10.70	60

Peak		Background			
10.40 to 10.60 $^{\circ}2\theta$		Region A		Region B	
time secs.	counts	time secs.	counts	time secs.	counts
120	60,332	60	28,784	60	28,506
120	59,870	60	28,943	60	28,368
120	60,105	60	28,634	60	28,204
Average	60,102		28,787		28,359

$$N = 28,787 + 28,359 = 57,146$$

$$\sigma = \sqrt{57,146} = 239 \quad 3\sigma = 717$$

$$N + 3\sigma = 57,146 + 717 = 57,863$$

The actual number of counts obtained for the integrated peak intensity was 60,102; therefore, the "suspect" peak is statistically present in the scan.

Standard Preparation

Optimal instrument conditions must first be determined with the use of tremolite standards: 1.0%, 0.75%, 0.5% tremolite by weight, prepared in a standard talc which is free of interfering peaks in the 11.0 to $10.0^{\circ}2\theta$ region.

Weigh out appropriate amounts of standard talc and tremolite both of which have been ground to pass a 325-mesh sieve. Transfer to a Waring Blender.* Add 100 ml of ethanol to the blender and blend at low speed for 5 minutes.

Carefully transfer the contents of the blender, with repeated ethanol washings, into a large beaker. Evaporate the ethanol on a steam bath.

Shake the sample in a plastic vial for 5 minutes on a Spex Mixer/Mill* to remove clumps and caked sample resulting from the evaporation of ethanol.

Determine by microscopy the homogeneity of the prepared standard previous to the x-ray diffraction analysis.

Press the homogeneous standard into a $1\frac{1}{4}$ " pellet with a backing of boric acid. Transfer $2 (\pm 0.2)$ g of standard to the die-holder and evenly distribute on a polished, scratch-free die. Distribute $4 (\pm 0.2)$ g of boric acid evenly on the talc layer. Press the mixture into a pellet under conditions suitable for obtaining a smooth planar surface (for example, a pressure of 15,000 to 24,000 lb calculated on a 3" ram has been found to produce suitable pellets). The resulting pellet must have a talc face which is free of flaws; if not, the pellet must be discarded (Note 3). Prepare two acceptable pellets from each standard.

*Registered Trademark

Sample Preparation

Prepare two pellets from each sample in the manner described for the standard pellets. Make a qualitative scan from 4 to $50^\circ 2\theta$ on one of these pellets to ascertain the presence of amphibole above the 2% level or the presence of mineral impurities having interfering peaks in the 11.0 to $10.0^\circ 2\theta$ (8.85 to 8.04\AA) region of the scan. The presence of such interference will eliminate use of the x-ray diffraction method for the sample, and one will have to proceed directly to the microscopical procedure.

Instrumentation

Instrumental variables are optimized on the 1% standard. Lower standards are then analyzed under the optimum conditions to determine the lower level of detection. Of major importance in obtaining maximum instrument sensitivity are a slow diffractometer speed combined with compatible recorder speed, and high attenuation combined with a statistically acceptable time constant on the ratemeter. Under appropriate instrumental conditions the peak obtained for the 0.5% standard should be detectable above background noise as shown in Figure 2.

Typical instrumental conditions employed for the Siemens Diffractometer (Model No. M386-X-A4), and Counter and Recorder Unit (Type T) are:

Radiation: Cu with K_α filter at 40KV and 24 ma
Divergence slit: 1° Receiving slit: 0.2 mm
Goniometer speed: $1/10^\circ 2\theta/\text{minute}$
Recorder speed: 300 mm/hour
Attenuation: 1×10^3 impulses/second
Time constant: $T(s) = 4$

Statistical error of 1.1% under these conditions

Rise Time = 0.18
Attenuator = 20

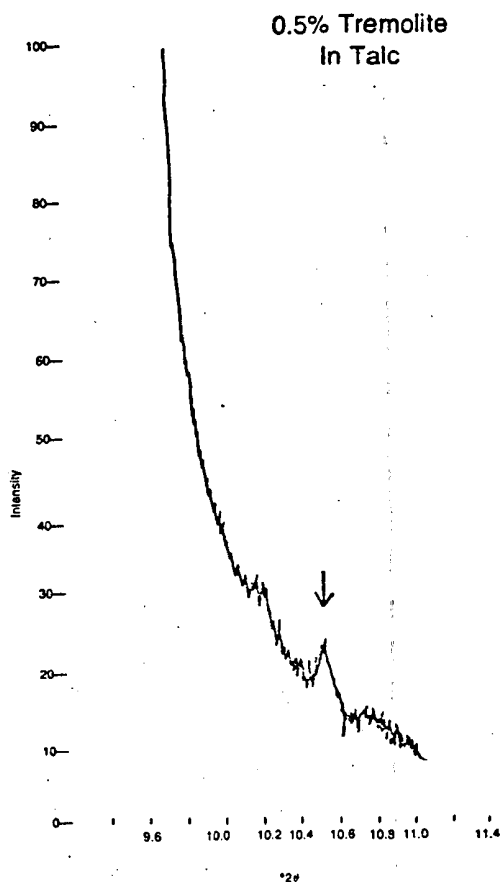


Figure 2

X-Ray Diffraction Scans

Place the standard or sample pellet in a suitable holder and slowly scan between 11.0 and 10.0°2θ. Then rotate the pellet 90° with respect to its original position in the goniometer and rescan between 11.0 and 10.0°2θ since pellet orientation may affect peak intensity. The presence of a reproducible peak (or peaks) is due to the presence of amphibole mineral (or minerals); the absence of peaks in this region indicates the absence of amphibole in the sample, within the limit of detection of this technique.

Report results as "None detected" or as "Detected at approximately X% level," where "X" equals the level detected.

Part II: Asbestiform Amphibole Minerals by Optical Microscopy and Dispersion-Staining

Apparatus

1. Polarizing microscope. Best results will be obtained if the instrument includes the following:
 - a. Individually centering objectives
 - b. Bertrand lens
 - c. High-intensity light source
 - d. Centering condenser/substage
2. Dispersion-staining device (Note 4)
3. Vacuum filtration equipment, including either a porcelain cone with glass fiber filter mat or a porous glass bottom cup

Reagents

1. Hydrochloric acid, 10% v/v
2. Cargille immersion liquid Series HD, $n_D^{25} = 1.605$ (Note 5)

Procedure

Acid Treatment

Because of the interference caused by some carbonates (e.g. calcite) in the detection of asbestiform amphiboles in talc by optical microscopy/dispersion-staining, it is necessary to first remove these carbonates by a simple acid leaching procedure:

Weigh out 2 g of the talc into a 100 ml beaker. Add 25 ml of 10% v/v HCl slowly (to prevent excessive evolution of gas if carbonates are present) and heat, with occasional stirring on a steam bath for 30 minutes.

Filter with vacuum filtration equipment, and wash several times with hot water. Dry the talc.

Optical Microscopy and Dispersion-Staining

Carefully disperse 0.1 mg of talc in one drop of Cargille HD liquid, $n_D^{25} = 1.605$, and cover with a clean cover slip.

Examine the sample in the dispersion-staining central stop mode. The substage diaphragm should be almost completely closed, the field diaphragm may be partially closed to enhance color contrast, and the polarizer should be in position.

Tremolite, actinolite and presumably other amphibole minerals, under these conditions, will show the following dispersion-staining colors: yellow changing to blue with rotation of the sample relative to the polarizer or yellow changing to orange with rotation. The variation of the color change is due to the fact that the tremolite may lie in one of two positions relative to its principal optical orientation.

Examine the sample for asbestiform fibrous amphibole minerals.

In order for an amphibole mineral to be considered asbestiform fibrous it must meet the following OSHA definition (Reference 4).

1. Particles must appear to be fibrous rather than as crystals or slivers.
2. The maximum diameter of a fiber to be counted is 3 microns.
3. The maximum length of a fiber to be counted is 30 microns.
4. The length to width ratio must be 5 or more to 1, that is, 5 times or more longer than wide.
5. The separate or individual fibers must contain fibrils or the "bundle of sticks" effect, unless they are at a nondivisible stage. A fibril cannot be subdivided and would be counted, if it meets the other criteria. The length to width ratio of 5 or more to 1 is not meant to imply that other particles are not hazardous.

Report results as "Asbestiform Amphibole Present" or as "Asbestiform Amphibole Absent."

It is imperative that both dispersion-staining color *and* fibrous morphology criteria be satisfied before identifying a particle as asbestiform amphibole, since other substances may show colors similar to those described.

Notes

1. Talc to be analyzed and the tremolite used to prepare standard samples must be finer than 325 mesh (maximum particle size of 44 microns). The Tekmar Analytical Mill (Model A-10) is recommended. It is available from:

Tekmar Company
P.O. Box 37202
Cincinnati, Ohio 45222

2. It is important that the homogeneity of the prepared talc-tremolite standard samples be verified by optical microscopy.
3. This requirement is critical since excessive surface scatter will cause abnormally high background counts.
4. The only commercially available dispersion-staining device is sold by:

Walter C. McCrone Associates, Inc.
2820 South Michigan Avenue
Chicago, Illinois 60616

5. Available from:

R. P. Cargille Laboratories, Inc.
Cedar Grove, New Jersey 07009

—or from laboratory suppliers.

References

1. Rohl, A. N., Langer, A. M., *Environmental Health Perspectives* 9, 95 (1974)
2. Rubin, I. B., Maggiore, C. J., *Environmental Health Perspectives* 9, 81 (1974)
3. L. S. Birks, *X-Ray Spectrochemical Analysis*, pages 54-55, Interscience Publishers (1959)
4. "Tremolite and Talc." U.S. Department of Labor, Occupational Safety and Health Administration, Field Information Memorandum # 74-92. November 21, 1974.

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Exhibit 140



J&J Consumer Companies Worldwide Specification

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ANALYSIS OF POWDERED TALC FOR ASBESTIFORM MINERALS BY TRANSMISSION ELECTRON MICROSCOPY

Name
TM7024

Type
Test Method

Revision	1	Owner	Corporate
Issued Date	1995-08-21	Expiration Date	9999-12-31
Geographical Scope	Local	Specification Category	Permanent
Security Classification		Review Interval (Months)	0

Related Information

Template	Test Method Global	SCO
Co-Owners		Owning Region
		North America

Revisions

Name	Rev	State	Description of Change	Reason for Change	Owner	Issued Date	Expiration Date
TM7024	1	Issued			Corporate	1995-08-21	9999-12-31

Approvals

Signer	Role	Organizations	Date/Time
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Content

Name	Format	File Size
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Reference Documents

Name	Description

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Related Specifications

Name	Type
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Document No.: TM7024

Franchise:

Location: ROYSTON, FLUID, KOLMAR

Document Type: Permanent

Expiration Date: None

Subject: ANALYSIS OF POWDERED TALC FOR ASBESTIFORM MINERALS BY TRANSMISSION ELECTRON
MICROSCOPY

<u>REVISION</u>	<u>AUTHORIZATION</u>	<u>DESCRIPTION OF CHANGE</u>
03/08/89	BCR011362	New Test method.
03/21/95	CR020127	Location revised. (Spec. Dept.)
08/21/95	CR020688	Location revised. (Spec. Dept.)

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Subject: ANALYSIS OF POWDERED TALC FOR ASBESTIFORM MINERALS BY TRANSMISSION ELECTRON MICROSCOPY

1.0 SCOPE & PURPOSE

This method is applicable to the identification and quantitation of small (typically 1-20 micrometer) asbestiform minerals in powdered talc. Samples may be previously screened with light microscopy or x-ray diffraction techniques.

2.0 PRINCIPLE OF METHOD

The combined techniques of transmission electron microscopy (TEM), selected area electron diffraction (SAED) and energy dispersive x-ray analysis (EDXRA) permit the detection of asbestiform minerals based on morphological characteristics, followed by a definitive mineralogical identification of each fiber.

3.0 INTERFERENCES

Interferences are caused by fibrous particles which must be distinguished from positively identifiable asbestos, and by large particles or particle aggregates which may obscure fibers. Positively identified non-asbestos fibers include rolled talc, ribbon talc, antigorite, silica fibers and iron oxide fibers. Organic additives such as perfumes may crystallize out as fibers or needle-shaped crystals in finished cosmetic products. In the absence of positive identification, all other fibers must be classified as unidentifiable.

4.0 INSTRUMENTAL CONDITIONS

The talc specimen grids are examined in the TEM at an accelerating voltage of 120 kv and at magnification of 20,000X and 5,000X.

5.0 SENSITIVITY

This method is capable of detecting a single fiber as small as 1 micrometer (mm) long by 0.075 mm wide in the entire TEM field, which results in a theoretical detection limit of 10^{-5} weight percent. Such fibers usually can be identified readily by SAED and EDXRA. The mass of a fiber with the above dimensions is 1.1×10^{-14} g for chrysotile and 1.5×10^{-14} g for amphibole.

6.0 LIMIT OF QUANTIFIABLE DETECTION

The detection of five or more asbestiform minerals of one variety in an analysis constitutes a quantifiable level of detection. When no asbestiform minerals are detected, a representative fiber size is used to calculate a detection limit. A representative fiber size is 3 mm long by 0.2 mm wide by 0.06 mm thick, which is considerably larger than the smallest fiber that can be detected (see section 5, SENSITIVITY), but is more typical of small asbestos fibers that are detected in talc analyses. The mass of five such fibers is calculated as follows:

$$\begin{aligned} 3 \text{ mm} \times 0.2 \text{ mm} \times 0.06 \text{ mm} &= 0.036 \text{ mm}^3 \text{ per fiber} \\ \times 3.3\text{E-}12 \text{ g / mm}^3 &= 1.2 \text{ E-}13 \text{ g per fiber} \end{aligned}$$

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x 5 fibers = 6E-13 grams per 5 fibers.

The limit of quantifiable detection for most talc analyses is approximately 6×10^{-4} weight percent. The theoretical and quantifiable detection limits assume homogeneity of the material being sampled.

7.0 QUALITY ASSURANCE

Blank suspensions are routinely prepared and tested in order to monitor potential residual contamination from the sample jars. Blank carbon-coated grids are routinely tested to monitor the ambient fiber count. If greater than 4 fibers per grid are present, the jars are pre-cleaned or new carbon-coated grids are prepared, respective of the test.

8.0 BACKGROUND CORRECTION

As of the time of this writing, background correction has not been necessary. The amount of background asbestos detected has been insignificant in comparison to the levels of asbestos found in contaminated samples.

9.0 PREPARATION AND ANALYSIS TIME

Preparation time per sample (including preparation of related materials) is one hour. Analysis search time per sample is a maximum of two hours.

10.0 APPARATUS

- 10.1 Analytical balance with 0.0001 gram sensitivity
- 10.2 Weighing boats
- 10.3 Narrow spatula
- 10.4 Wide mouth polyethylene jars (125 ml)
- 10.5 Mild ultrasonic bath, minimum 50 watts
- 10.6 Micropipettor (5-10 ml range) with disposable tips
- 10.7 Standard 3 mm diameter, 200 mesh, copper TEM grids, covered with a carbon-coated formvar film.
- 10.8 Transmission electron microscope (TEM) with an 80-120 kv accelerating voltage and energy dispersive x-ray analyzer.

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11.0 REAGENTS

11.1 Methyl cellulose, powder, USP 4000 cps - Fisher Certified Reagent #M-352 or equivalent

11.2 Water: deionized, particle free (+0.2 mm filtered)

11.3 Methyl cellulose solution: 0.002% (wt/vl) (20 ppm). Dissolve 20 % 0.5 mg of methyl cellulose in 500 ml of deionized particle free water to make a 0.004% stock solution. Dilute 1:1 to make a working solution.

NOTE: Methyl cellulose acts as a wetting agent to aid in maintaining a uniform particle distribution as the sample dries, by greatly reducing the surface tension of water.

12.0 SAMPLE PREPARATION

12.1 Transfer 30 to 50 mg of talc powder to a clean 125 ml polyethylene jar.

12.2 Add 80 ml of 20 ppm methyl cellulose solution, cap and shake vigorously for one minute.

12.3 After shaking, loosen cap and ultrasonicate for 10 minutes in order to disperse the finer particles. Then shake again for one minute to produce a uniform suspension.

12.4 Immediately after shaking, uncap and remove 9.2 microliters with a micropipette.

12.5 Transfer a 9 ml drop to a carbon film covered TEM grid. (Grid was first lightly anchored by 2 parallel strips of double-stick tape mounted 3 mm apart on a clean glass microscope slide.) Repeat to make two sample grids per talc sample.

NOTE: Do not expel the remaining 0.2 ml suspension from the micropipette tip. It tends to sputter and frequently destroys the stability of the sample drop.

12.6 Transfer slide with grids to a desiccator. (Drying time is 2-3 hours.) Do not leave the grids on the slide for more than one day as the double-stick tape may adhere too tightly.

NOTE: The talc:water ratio may need to be varied for some samples. Preparation of talc samples with a significantly finer or coarser particle size results in large differences in particle coverage on the TEM grid.

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Document Type: Permanent

Expiration Date: None

Subject: ANALYSIS OF POWDERED TALC FOR ASBESTIFORM MINERALS BY TRANSMISSION ELECTRON MICROSCOPY

13.0 TEM ANALYSIS

- 13.1 Definition of fiber: An elongated particle with parallel sides and an aspect ratio K3:1. The definition employed may vary with the needs of the client.
- 13.2 Scan sample at 120-150X magnification to check for even dispersion of particles and to locate grid squares with optimum particle density. (Optimum particle density is particle coverage over 15-35% of the field of view.)
- 13.3 Scan three grid squares on each grid at 20,000X magnification and seven grid squares on each grid at 5,000X for asbestiform minerals. Each asbestiform mineral is recorded as to type (chrysotile, tremolite, anthophyllite, etc.), structure (bundle, clump, fiber) and dimensions (length x width).
- 13.4 Questionable fibers are examined first by SAED. The chrysotile SAED pattern is unique and diagnostic. Amphibole SAED patterns are variable but usually characteristic. Additional analysis and measurement of amphibole SAED patterns are done if warranted.
- 13.5 Ten percent of chrysotile fibers are checked by EDXRA for further confirmation. If the SAED pattern is not clearly diagnostic, or if it is consistent with an amphibole SAED pattern, then it is examined by EDXRA to confirm the identification or to identify the type of amphibole.

14.0 CALCULATION OF RESULTS

- 14.1 Mass of chrysotile fibers: $M(f)$
 $M(f) = \pi r^2 l \times d$
 $\pi = 3.14159$
 r = fiber radius
 l = fiber length
 d = density of chrysotile = 2.55×10^{-12} g/mm³
- 14.2 Mass of asbestiform amphibole particles: $M(a)$
 $M(a) = l \times w \times th \times d$
 l = length
 w = width
 th = thickness Z 0.3 width (approximation)
 d = density of amphiboles = 3.3×10^{-13} g/mm³
- 14.3 Mass of talc deposited on each TEM grid: $M(s)$
 $M(s) = T \times (V/H)$
 T = amount of talc sampled (step 12.1)
 V = volume of aliquot transferred to TEM grid (step 12.5)

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H = volume of methyl cellulose solution (step 12.2)

14.4 Total estimated talc mass examined: $M(t)$

$M(t) = M(s) \times (N \times A(s))/A(g)$

N = number of grid squares examined

A(s) = area of a single TEM grid square

A(g) = area of an entire TEM grid (effective area over which a 9 microliter drop of suspension dries)

14.5 Weight percent:

$$\frac{\text{sum total of } M(f) \text{ or } M(a) \times 100}{M(t)}$$

15.0 CALCULATION OF A DETECTION LIMIT

15.1 $M(dl)$ = A minimum quantifiable mass of asbestos fibers, based on the detection of 5 fibers (approximately 6E-13 grams, from Section 6).

15.2 Detection Limit (Weight Percent) = $\frac{M(dl) \times 100}{M(t)}$

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Exhibit 141

COLORADO SCHOOL OF MINES RESEARCH INSTITUTE

A Procedure to Examine Talc for the Presence of
Chrysotile and Tremolite-Actinolite Fibers

Prepared for

Johnson & Johnson
501 George Street
New Brunswick, New Jersey


By

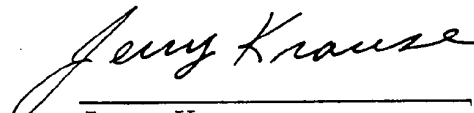
Colorado School of Mines Research Institute
Golden, Colorado

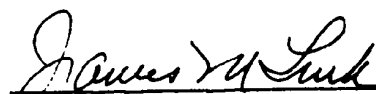
Project C10704

December 27, 1973

APPROVED:


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COLORADO SCHOOL OF MINES RESEARCH INSTITUTE

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INTRODUCTION

The purpose of this document is to report the methods used at the Colorado School of Mines Research Institute for detection of chrysotile and/or tremolite-actinolite in samples predominantly composed of talc. The methods described herein have evolved over a period of time, with the aid of suggestions from many individuals, and are frequently subjected to review.

As the impurity level becomes very low ($\ll 1\%$), it is necessary to examine increasingly larger amounts of sample in order to detect the impurity. As a result of the requirement to detect the proverbial "needle in a haystack," we have evolved a procedure which preconcentrates the impurities prior to examination. The net effect is that a large initial sample is fractioned in order to reject the majority from further examination.

OBJECTIVE

The objective of this work was to develop a procedure to screen talc for the presence of chrysotile and tremolite-actinolite asbestos minerals. Based on past experience with detecting and identifying minerals when present at low levels, a concentration of the phases to be detected was considered essential to the success of any suggested procedure. Once concentrated the impurities could be detected by conventional methods of examination.

SUMMARY AND CONCLUSIONS

A procedure to detect the presence of chrysotile and/or tremolite-actinolite fibers in talc is presented. The procedure involves two heavy liquid separations to concentrate any chrysotile and tremolite-actinolite which may be present. The heavy liquid concentrates are examined by optical microscopy for the presence of optical size (greater than approximately 2 microns in length) fibers of chrysotile and/or tremolite-actinolite. The procedure is capable of detecting fibers present at a level of approximately 10 ppm or less.

DISCUSSION

DETAILS OF THE PROCEDURE

The optical and physical properties of talc, chrysotile, and tremolite-actinolite important to their separation, concentration, and identification are listed in the table on the following page.

The separation and concentration technique involves heavy liquid separations and is therefore dependent upon specific gravity differences. Identification of the phases thus separated and concentrated is based upon their optical and morphological properties. It is estimated that the following procedure will allow the detection of chrysotile and/or tremolite-actinolite when each is present at a level of approximately 10 ppm or less.

Samples

This method may be applied to a variety of samples ranging from raw ore to final metallurgical concentrates. Raw ore samples should ideally be crushed and sized to -200+325 mesh to liberate talc and other minerals. Metallurgical process samples containing a large proportion of -325 mesh material can be handled in the same manner although the centrifuging and filtering times will be increased.

Separation Details

Five-gram samples are added to each of two 125-ml separatory funnels which contain approximately 75 ml of heavy liquid (2.90 sp gr).⁽¹⁾

(1) Centigrav; commercially available from American Mini-Chem Co., Corapolis, Penn., 15108.

Relevant Optical and Physical Properties
of Talc, Chrysotile, and Tremolite-Actinolite⁽¹⁾

	Optic Sign	Optic Orientation	Refractive Indices			Specific Gravity	Morphology
			α	β	γ		
Talc	(-)	$\begin{cases} Z \wedge a \cong 10^\circ \\ X \cong b \end{cases}$	1.539-1.550	1.589-1.594	1.589-1.600	2.59-2.83	Plate Fiber ⁽²⁾
Chrysotile	(-)	X = C	1.532-1.549	--	1.545-1.556	~2.55	
Tremolite- Actinolite	(-)	$Z \wedge c = 10-21^\circ$	1.599-1.688	1.612-1.697	1.622-1.705	3.02-3.44	Fiber

(1) Data from Deer, Howie, and Zussman, Rock Forming Minerals, vol. 2, 1962; vol. 3, 1963.

(2) Fiber -- any material having a form such that it has a minimum length to average maximum width of 3:1.

Each sample is well dispersed by thorough shaking of the loaded stoppered funnels, and then centrifuged at 800 rpm for two intervals of 1/2 hr. The float material is agitated slightly between centrifuge intervals to aid in releasing high specific gravity particles which may be trapped in the tightly packed floating fraction. The heavy and light fractions are collected separately on 0.45 μ millipore filters, washed with ethanol or isopropyl alcohol, dried, and carefully weighed. The heavy fraction (sp gr >2.90) will be examined for tremolite-actinolite.

The light fraction (sp gr <2.90) collected above is reprocessed in an identical manner in a liquid of sp gr 2.65. The light fraction with sp gr <2.65 will be examined for chrysotile. The fraction with sp gr >2.65 and <2.90 is assumed to be predominantly talc and therefore is not subjected to further examination. This fraction could of course contain fragments of other minerals locked to the talc.

The 2.65 sp gr liquid is prepared by diluting Certigrav 2.90 sp gr liquid with n, n dimethylformamide having a specific gravity of 0.95. The heavy liquid can be recovered from the alcohol-n, n dimethylformamide washings by extraction with large volumes of water.

The fractions recovered from the heavy liquid separations generally amount to 20 mg or less.

Microscopy

Optical examination of the heavy liquid separates for the presence of fibers is a sensitive examination method. Optical microscopy can detect fibers with a length greater than approximately 2 μ , when present at a level

of approximately 0.1% or greater. If optical examination at magnifications up to approximately 625X does not reveal the presence of fibrous particles, the sample can be passed as being clean. If fibrous material is detected optically, then specific identification of the fibers must be attempted. Optical identification is difficult and subject to numerous errors, especially when working with small particles which are near the resolution limit of the microscope. Electron microscopic examination employing selected area electron diffraction and/or x-ray emission spectrography may be required in order to specifically identify small fibrous particles.

The following optical identification schemes require a great deal of expertise, and are subject to errors introduced by small particle size, the presence of talc fibers, plates lying on edge thereby appearing to be fibers, overlap in optical properties, and variable reaction of chrysotile to the iodine stain.

Tremolite-Actinolite

The heavy liquid separate having a sp gr >2.90 is mounted in immersion oil of refractive index 1.600 for transmitted light examination under a petrographic microscope. All amphiboles have refractive indices appreciably greater than 1.600 and will be readily visible. All observations are made at magnifications of 125X and 250X. Single particles are occasionally examined at a magnification of 625X. Tremolite-actinolite fibers are identified by having length to width ratios greater than or equal to 3:1; refractive indices greater than or equal to 1.600; and extinction angle varying between 10° and 21°.

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Special Talc Studies

Monthly Report for March, 1974

Assay Methods for Asbestos Minerals in Talc

Methods developed by Prof. Reynolds (Dartmouth), for Windsor Minerals were tested by the Analytical Division (P. Carpe report, March 11, 1974). The methods separate and concentrate the heavy (tremolite-actinolite) and light (chrysotile) forms of asbestos by heavy liquid flotation. Detection and identification is by X-ray scan (chrysotile), or optical microscopy (tremolite-actinolite). According to this report, the method for chrysotile is very difficult to carry out and not practical on a day to day basis. However, the method for tremolite-actinolite is good and has a detection limit of 0.02% using optical dispersion staining as the preferred method of identification.

Analytical will also test a method developed by Dr. Pooley for detection of asbestos in talc. The necessary equipment is being sent by Dr. Pooley from England.

Following the allegations of silica in Johnson's Baby Powder through a NIOSH Report, Messrs. Schelz, Davies and Rolle have met with representatives of the NIOSH laboratory which generated the analyses and report. They admitted that their analytical method (wet analysis) may be inaccurate and the determinations would be expected to be on the high side.

The powder samples used in their study have been given to us and will be analyzed by X-ray diffraction and DTA. An official Johnson & Johnson rebuttal will be prepared to repute the NIOSH analyses.


Exhibit 143

Johnson & Johnson

New Brunswick, N.J.

March 11, 1974

Subject: Methods of Concentration of
Asbestos in Talc-Project #0503-00

Dr. J. P. Schelz 
to
Dr. F. R. Rolle

I have tested the heavy liquid flotation methods described by Dr. Reynolds. Both procedures are effective in concentration asbestos fibers in talc, but neither would provide a useful means of quantitation, since there is a carry-over of 5-10 percent of the talc in the concentrates.

The procedure for "heavy" fibers (actinolites and tremolite) permits the detection of asbestos at original concentration as low as 0.02 percent by weight, while the existing procedures (step-scanning x-ray diffraction) are effective to 0.2 percent. The dispersion staining technique of optical microscopy was employed to detect tremolite fibers in the concentrates. The method is straightforward and rapid requiring about 20 minutes of operator time and about one hour overall. If removal of carbonates is desired these times become 30 minutes and two hours, respectively.

The method for chrysotile is somewhat more difficult to perform, if Dr. Reynold's description is followed explicitly. This method employs an aqueous dispersion with surfactant to remove the fine fibers (less than 2 um) and then heavy liquid flotation for the larger fibers. The procedure works as described but the removal of the fine fibers from the aqueous medium is quite difficult since they will not centrifuge and tend to clog most filter media fine enough to catch them. If however, the aqueous dispersion is omitted and only the heavy liquid separation employed, samples containing 0.2 percent chrysotile can be concentrated to the extent that the chrysotile can be detected by differential thermal analysis.

- 2 -

There is another method, developed by Dr. F. Pooley, which is as effective for chrysotile as that of Dr. Reynolds and will simultaneously concentrate the heavier forms of asbestos. I plan to test the method as soon as I receive the apparatus which Dr. Pooley is submitting.

Philip Carpe
Philip Carpe

kd

cc: Dr. E. R. L. Gaughran
Dr. A. J. Goudie
Mr. G. Lee
Dr. W. Nashed
Dr. D. R. Petterson
Dr. T. H. Shelley
Mr. V. Zeitz, Windsor Minerals
Central File
Section File

JNJ000250919

Metadata

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PgCount	2	ORIGINAL

Exhibit 144

cc: Dr. A. J. Goudie
Mr. G. Lee ←

Johnson & Johnson

New Brunswick, N.J.
November 26, 1974

Subject: REVIEW OF EXPERIMENTAL TECHNIQUES
FOR THE CONCENTRATION OF ASBESTOS
MINERALS IN TALC - PROJECT #0503-00

file talc ANALYTICAL
RECEIVED

NOV 27 1974

Dr. F. R. Roile

G. LEE

Our preliminary investigation of experimental techniques for the concentration of asbestos minerals in talc has been in two areas;

1. The concentration of both amphibole and serpentine asbestos minerals from talc by the flotation procedure suggested by Dr. Fred Pooley. In this technique 25 gm. samples of talc are floated in a 3 l. flotation cell. Advantage is made of the natural floatability of talc in separating and concentrating the asbestos minerals to the extent of about eight times the original concentration by a single flotation step. The process can be repeated several times on the talc tailings to give a final concentration of the asbestos minerals of about twenty times the original amounts.

After some initial problems in having the flotation cell built, we have encountered some difficulty in rapid, fine filtering of 2-3 l. of solution to obtain the tailings which include any asbestos minerals. Contrary to Dr. Pooley's procedure, we feel that dry, homogeneous talc standards containing tremolite and chrysotile should be prepared for the purpose of developing the concentration technique, rather than admixing the asbestos minerals directly with the talc in the flotation cell. This concentration process shows promise after refinement of technique.

2. The concentration of actinolite/tremolite and chrysotile from talc by individual heavy liquid separation techniques developed by Dr. Robert Reynolds, Dartmouth College. Dr. Reynolds, of the Department of Earth Sciences, had been requested by Mr. V. Zeitz of Windsor Minerals to work on an actinolite concentration technique. This method utilizes the difference in densities between actinolite (and other amphiboles) and talc to effect separation in a heavy liquid medium.

Following Dr. Reynolds procedure, we have been able to detect tremolite by optical microscopy/dispersion staining in the separated fraction from a sample containing initially as little as 0.01% by weight tremolite in Vermont talc. Dr. Reynolds was then asked to develop a similar method for the concentration of chrysotile from talc. Using this separation technique, no chrysotile could be detected by DTA in the concentrate from a talc standard containing 0.01% by weight chrysotile. A long and impractical

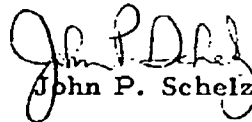
J&J-0084587

-2-

November 26, 1974

final filtration is required because of the extremely fine nature of the chrysotile.

Each of these experimental techniques warrants additional work. We have not been able to pursue the concentration of asbestos minerals from talc because of a shortage of personnel and the low priority which had been set for the project.


John P. Schelz

gm

J&J-0084588

Exhibit 145

TF
PROPOSED SPECS FOR ANALYZING
TALC FOR ASBESTOS

127023
Johnson & Johnson

F

New Brunswick, N.J.
May 16, 1973

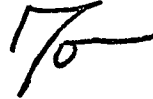
Subject:

Dr. F. R. Rolle

I am going to England Friday, May 25. I have been asked to bring along our proposed specs for analyzing talc for "asbestos."

Please get me copies of all reports, correspondence, etc., that are pertinent, plus a cover memo outlining our recommendations.

England is considering method of preconcentrating the asbestos so as to be able to analyze by X-ray. They find no "asbestos" by doing this with Italian talc. They find (Pooley) 0.05% of a tremolite-type in Vermont.



T. H. Shelley

mf

c: Dr. R. A. Fuller
Dr. A. J. Goudie
Dr. W. Nashed
Dr. D. R. Petterson

RECEIVED
MAY 18 1973
W. NASHED
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Johnson & Johnson

New Brunswick, N.J.
May 22, 1973

Subject: PROPOSED SPECS FOR ANALYZING
TALC FOR ASBESTOS

Dr. T. H. Shelley

I. USP

II. Other Methods

Step Scanning X-Ray Diffractometry

Advantages

Disadvantages

Preconcentration of Asbestos (Pooley Method)

Differential Thermal Analysis

Microscopy

Electron Microscopy and Petrology

Dispersion Staining

III. Present Strategy

F. Robert Rolle

F. Robert Rolle, Ph.D.

ab

cc: Dr. A. J. Goudie
Dr. G. Hildick-Smith
Dr. W. Nashed ✓
Dr. D. R. Petterson

RECEIVED

MAY 23 1973

W. NASHED
JOHNSON & JOHNSON

- 1 -

I. USP

We have been working on a preliminary draft with Mr. George Heinze on developing a USP method for the detection of asbestos in talc. Exhibit A is the USP XIX comment proof on X-ray diffraction. Exhibit B is our detailed procedure which has been submitted to Mr. Heinze, for determination of amphibole (such as, tremolite) and serpentine (such as, chrysotile) in talc by scanning X-ray diffractometry. Using this method on Italian Talc used in SHOWER TO SHOWER* Powder, we find a level of detectability of 1% for Tremolite and 5% Chrysotile.

II. Other Methods Which Have Been or Are Under Consideration
for the Detection of Asbestos in Talc

Step Scanning X-Ray Diffractometry

Advantages: Level of detectability better than by scanning X-ray diffraction. For example, by this method we can detect 0.1% tremolite and 3% chrysotile in Italian talc (Exhibit C).

Disadvantage: Using the step scanning procedure, it takes one day per sample for analysis vs. a small fraction of a day for the scanning method.

*A Trademark of JOHNSON & JOHNSON.

- 2 -

Preconcentration of Asbestos followed by X-Ray Diffraction
Analysis (Pooley Method)

Dr. Pooley has developed two techniques for preconcentration of chrysotile and tremolite in talc followed by X-ray diffraction analysis. For chrysotile (Exhibit D), his level of detectability is 0.05% and when this method is applied to Italian and Vermont talc, no chrysotile is detected. The second technique developed also by Dr. Pooley involves preconcentration of tremolite in talc (different procedure) followed by X-ray diffraction analysis.

This technique has not been written up yet, but evidently when applied to Vermont talc, 0.05% of tremolite-type is found. The limitation of this method is that it may be too sensitive.

Differential Thermal Analysis (DTA)

DTA has proven to be a relatively fast and sensitive method (at least 1%) for detection of chrysotile in talc (Exhibit C). The DTA method is not applicable for the detection of tremolite in talc. At our suggestion, the FDA recently purchased a DTA unit, presumably to look into this method for detecting chrysotile.

- 3 -

Microscopy

A. Electron Microscopy and Petrology

The areas of electron microscopy and optical microscopy (petrology) have been thoroughly evaluated, but though, not without merit, they suffer from the following limitations:

- a) require a fair degree of expertise
- b) in the case of electron microscopy, we are dealing with an expensive instrument that few laboratories have.
- c) one is viewing a very small amount of material (μg) under the microscopy and one wonders how representative it is of the bulk material. Multiply sampling and viewing under the microscopy may eliminate this problem, but it results in consumption of a great deal of time.
- d) the level of detection really depends upon the amount of time spent with the microscope.
- e) quantification by particle counting is very time consuming and normally not done.

B. Dispersion Staining

The dispersion staining method championed by Dr. Walter McCrone looked initially very exciting as a quick, easy method for scanning talc for asbestos. However, it was found (Exhibit E) that certain non-asbestos minerals gave the same dispersion staining characteristics as the asbestos minerals. The method evidently lacks specificity when applied to talc.

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III. Present Strategy

Present plans call for scanning X-ray diffraction for the detection of both amphibole and serpentine asbestos in talc. In the case of chrysotile (serpentine), Differential Thermal Analysis may be a good alternate method since it offers a level of detectability of 1% chrysotile in talc vs. 5% chrysotile in talc by scanning X-ray diffraction.

Exhibit 146



JOHNSON & JOHNSON
LIMITED

CTF Talc
ANALYTICAL

SOUTHAMPTON ROAD COSHAM PORTSMOUTH HANTS. PO6 4RL TEL: COSHAM 75298

for 60 years
the most trusted name in Britain for
Baby Products and Surgical Dressings

Our ref: IWS/VW

18th February 1975.

Dr. R. Rolle.,
Johnson & Johnson Research Centre.,
U.S. Highway No. 1.,
North Brunswick,
New Jersey 08903.,
U. S. A.

Dear Bob,

I am writing to put you in the picture regarding the U.K. work on analytical methods and to ask you to keep me informed on your front so that we can present a united front on this topic. I understand that you are the Chairman of the C.T.F.A. Committee on analytical methods. I serve on the T.P.F. Talc working party and the T.P.F. analytical sub committee.

I am therefore enclosing data on an Infra Red technique being worked on by Yardley and Avon. This is intended to be specific for tremolite and to be used only as a routine quality control check. Perhaps you could let me have your comments on the I.R. technique together with any results you have obtained in your own researches on Infra Red techniques.

I have also enclosed our test method for the proposed Xray technique which was drawn up by Boots Ltd in conjunction with Dr. Pooley. We deliberately have not included a concentration technique as we felt it would not be in worldwide company interests to do this. However, Fred tells me that you are now considering such a test in the U.S.A. If you are, it is important that the U.K. uses the same technique.

We have started a reference library of standard talc/minerals and I will send you a photocopy of the data so far. We would be pleased to include any input you might feel necessary.

Avon, U.K., have been receiving copies of the CTFA task force meeting minutes. Could I ask you to provide me with copies also?

Many thanks for your help.

Kind regards,

Yours sincerely,

J. W. S. G. A. G.

J. W. S. G. A. G.

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Exhibit 147

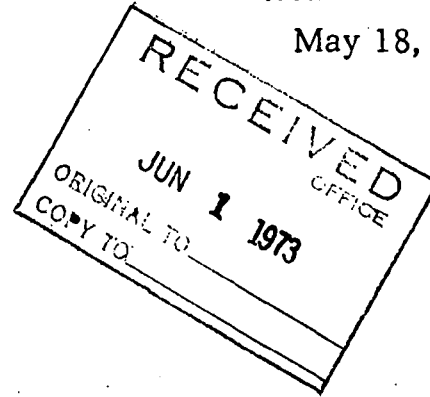
Johnson-Johnson

LCVZ - m

New Brunswick, N.J.

May 18, 1973

Subject: Talc Symposium - May 8, 1973
Department of the Interior



Mr. W. Ashton
Dr. A. Goudie to Dr. R. Rolle
Dr. G. Hildick-Smith
Dr. D. Petterson
Dr. W. Nashed
Dr. T. H. Shelley

The symposium was opened by Mr. D. P. Schlick, Deputy Director for Health and Safety of the Bureau of Mines. He stated that the Bureau of Mines was interested in the situation where dust other than silicates can cause diseases, and that this symposium had been specifically convened to consider the occupational exposure to talc, and what safety standards were necessary for the mining and milling of talc. He said the Bureau of Mines was interested in the physical properties rather than the chemical properties of such dusts and that they recognized that a talc known as fibrous talc (which he defined as one containing tremolite and/or amphibole asbestos materials) existed, but that asbestos would be considered only as it relates to talc. He also stated that written communications addressed to the Bureau received prior to June 1 will be considered part of the symposium.

The next speaker was Mr. Arthur P. Nelson, Acting Assistant Director of the Bureau of Mines. He stated that TLV's as recommended by the ACGIH are presently used as limits by the Department of Mines, but that there may be standards set in the future that will not be derived from ACGIH recommendations. He then introduced Dr. Goodwin who as Chief of the Division of Health for the Bureau of Mines was the moderator for the symposium. Dr. Goodwin introduced the first speaker, Dr. Morris Kleinfeld as a consultant and expert on talc exposure in New York State. Dr. Kleinfeld said that he was interested in the mortality of talc miners and millers in New York State and with making clinical correlations to the exposure to talc dust of such miners and millers. Dr. Kleinfeld said that he had been studying the mortality of talc workers since 1940 including all those with a minimum exposure of 15 years to talc mining and milling operations. The total number of workers included in the study is 260 among which there were 108 deaths over the period studied. His overall view was that there was some indication of a higher rate of pneumoconiosis when there was significant exposure over long periods of time to talc and other silicates such as tremolite and anthophyllite. In addition, for the whole group there was an increased mortality rate due to respiratory carcinoma in the age group 60 to 79, but not in the age group 40 to 59, which was a different result from that seen in asbestos workers. As far as gastro-intestinal malignancies were concerned talc

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miners showed no higher proportion than the general population, in contrast to asbestos miners, particularly those exposed to chrysotile, where the G-I rate was appreciably higher than for the general population. Dr. Kleinfeld stated that of the 108 deaths noted 25 occurred from malignancy which broke down as follows:

- 13 from lung and pleural carcinoma
- 9 from gastro-intestinal carcinoma
- 3 from other malignancies

The 13 respiratory mortalities are about 4 times that expected in a 60 to 79 age group. However, in the absence of smoking data and other tangential information a total explanation cannot be made. Dr. Kleinfeld then explained that the study had been broken down into six 5-year periods and that in the periods 1960 to 65, 1965 to 70 and updating of it through May 1973, that there was no difference in the expected (normal population) versus the observed rate in talc miners for cancer of the respiratory or G-I tract. This was not true prior to 1960. Kleinfeld's explanation is that he feels this is due to increased environmental controls instituted in the mines since 1960, although the exposure was still about 4 times above the TLV's for asbestos fibers. He feels that the present threshold level is satisfactory for asbestos and talc and that we still do not have definite findings for the rate of pneumoconiosis as a result of talc exposure. He stated that some workers exposed to 60 million particles per cubic foot versus a TLV of 20 for talc, even though mixed with tremolite and anthophyllite, had no higher rate of pneumoconiosis than the normal population.

The next speaker was Mr. Richard Lamar of Johns-Manville who spoke about talc from the viewpoint of a professional mineralogist. He stated that the medical aspects are the most important to be studied but that prior to this definitions of commercial talc and analytical methods of characterization have to be established. He stated that in talking about commercial talc versus talc mineral we have to speak of the associated minerals found in commercial talc, and that he felt the definition of fibrous talc should be defined as talc ores contaminated with asbestiform mineral rather than as presently defined. Since most commercial talcs contain higher proportions of particles under 5 microns which would not be counted in the present standard method, Lamar feels that X-ray diffraction is the most powerful and useful tool for the characterization of various talcs, with a limit of detection of 1% tremolite. He also stated that DTA was very useful, not particularly for tremolite, but for chrysotile. In concluding he warned that attempting to use optical microscopy alone and depending upon particles shape or morphology could be very misleading. He felt that electron microscopy was more reliable and revealing than optical microscopy but even here some mistakes can be made with talc platelets that are rolled up in tubes to look like fibers, as well as platelets on edge.

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Mulryan, the President of the United Sierra, gave the next presentation. He claimed that United Sierra was the largest commercial talc producer in the United States, and that their Montana mines produced non-asbestos containing talc. To illustrate this he had transmission electron microscope pictures of New York State materials showing tremolitic fibers but Montana material exhibiting only plates. He went on to say that United Sierra currently is not working any mines containing tremolite and that it meets the TLV's for the mineral and asbestos fiber standards. In addition they studied a group of 180 of their miners with 7 of them having between 25 and 44 years of service who were all in good health by present standards. The meeting was then turned over to Mr. Robert Bacon of Vanderbilt who introduced three speakers, preceding each with a motherhood and God introduction. The first speaker was Dr. C. S. Thompson, a mineralogist. He said he was going to confine himself to a clarification of the terminology of nomenclature associated with asbestos and talc. He stated that asbestos was a non-scientific term and that there are six minerals listed in the Federal Register as being asbestiform minerals. He claims that anthophyllite, tremolite and actinolite are non-asbestiform varieties and chrysotile, crocidolite and amosite are the only truly fibrous or asbestiform minerals that should be included in defining fibrous talc. In addition, he stated that when characterizing fibers you needed more than merely just an aspect ratio of 3 to 1, but you had to look at tensile properties and also make sure that these were not just fracture situations.

Dr. William Smith then spoke on his research of the last 8 years of exposure of animals to various mineral dusts. He says that during that time he and his coworkers have studied the fibrogenic and carcinogenic properties of various minerals. They chose the golden hamster as the species of choice to establish reactions to talc dust, which was done by making intrapleural injections into the hamsters. He stated that tremolite containing talc led to very few complications compared to the same experiments when carried on with the four principle types of asbestos. In addition, the asbestos led to the formation of mesotheliomas which was not true of tremolitic talc.

Incidence of Mesotheliomas
From Intrapleural Injection

<u>Dose</u>	<u>Chrysotile</u>	<u>Amosite</u>	<u>Anthophyllite</u>	<u>Crocidolite</u>
25 mg	9	-	-	-
10 mg	4	3	3	10
1 mg	0	0	0	2

The only reaction that he saw from tremolitic talc was some increase in fibrotic adhesions but even this was not to the extent that the asbestiform fibers caused fibrosis. In addition he stated that if you take chrysotile and mill it fine enough you do away with it causing mesotheliomas. He therefore stated that asbestos fibers that could be optically characterized and recognized as such had carcinogenic and fibrogenic potential but if the fibers were milled so they could no longer be recognized

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optically they no longer had a carcinogenic potential. In conclusion he stated that tremolitic talc was noncarcinogenic and had a very small fibrogenic potential compared to chrysotile which was carcinogenic and had a significant fibrogenic potential.

The last speaker representing Vanderbilt was Dr. G. W. H. Shepers of the Veterans Administration. Dr. Shepers gave the most extensive presentation of the day — about an hour and a quarter in length — in which he went into the pathology of dust caused lung diseases in great detail. He said that talc, meaning commercial talc with tremolite, had certain biological actions and could be compared in its biological responses to other forms of different minerals, some of them with fibrous aspects. Using a scale of 0 to 10 indicating increasing seriousness of response with higher numbers, he stated that on such a scale talc would rate 0 to 1 and be least injurious with tremolite coming next, then chrysotile with crocidolite being the most dangerous and having a rating of 9 to 10. He stated that tremolite is basically crystalline and not fibrous and does not have a biological toxic reaction when compared to a true asbestos fiber such as chrysotile. In addition he claimed that there was a dose response characteristic for the different type of minerals. This must be kept in mind and also that extremely large doses of talc were necessary to produce the onset of any reaction. In addition he then discussed the speed of reaction, saying that talc is retained in lung tissue for long periods of time but does no harm there, while chrysotile and other forms are eliminated faster, thus by the time the pathology is actually examined they will have done their harm but no longer be observable. Therefore the casual researcher will conclude that the talc was the causative factor when this in fact is not so. He stated in addition that talc is probably only responsible for no more than 5% of the physiological changes in the lung and that it does not lead to emphysema. In summary he said that tremolitic talc is relatively innocuous. When questioned what he meant by tremolitic talc he stated that this was talc with 5 to 25% of tremolite. He made a real effort to attempt a total whitewash for tremolitic talcs on behalf of Vanderbilt.

The last speaker to give a prepared presentation from industry was Mr. Harold Stanley of Pfizer. He spoke on the detection and identification of asbestos and asbestiform minerals in talc. Essentially his presentation was the same that he had privately communicated to us in a preprint at a meeting with us here earlier in 1973. He said that Pfizer has used light microscopy, transmission electron microscopy and X-ray diffraction to characterize various talcs and that by using step scanning X-ray diffraction techniques you could lower the limits of detection to a quarter percent tremolite and a half percent of chrysotile provided that chlorite is not present in attempting to determine the chrysotile. If chlorite is present he made the recommendation to go to electron microscopy with a standard that if no fibers are seen in a hundred fields of view by electron microscopy then we can consider the materials to be free of asbestos and asbestiform minerals. He further stated that if there were some doubt as to the presence of fibers because of the possibility that they might be rolled up platelets then

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electron diffraction should be used for positive identification. He also stated that in Pfizer's Montana talc no fibers of any sort were found. He recommended X-ray diffraction as a standard method with electron microscopy as a referee method, but felt that optical microscopy was actually a poor choice.

The first speaker to speak in favor of more stringent controls on talc was Dr. Arthur Langer of Mt. Sinai School of Medicine. He stated that his interest was in the identification and characterization of mineral bodies in human tissue and in consumer products. His thesis was that talc is not a specific mineral as the term is generally used but is a mixture of minerals that are finely ground and tend to be platey. He said that there are true talc fibers which can be identified by transmission electron microscopy. He further stated that so called commercial talc from New York is really a mixture of platey talc and asbestiform minerals mixed together. He claimed that in characterizing talcs, the methods of choice were X-ray diffraction, transmission electron microscopy, electron diffraction, optical microscopy and microchemical analysis. He referred to the same paper that Dr. Kleinfeld had spoken of earlier which he stated would be published in Environmental Research in June or July. However he had a different interpretation of the statistics quota. He said that 39 men had been examined who are miners and millers in New York State mines versus a group of 39 others as a control group which had not had exposure to talc dust. He claimed that of the 39 men in the exposure group only 1 had signs of pneumoconiosis as confirmed by X-ray but that 25% of the group presented clinical signs of pneumoconiosis. He stated that while no malignancies had been detected to date, the average exposure of this group was only $15\frac{1}{2}$ years and that another 10 years would have to transpire before we would see the onset of malignancy. His conclusion therefore was that talc is not innocuous and that all talcs as far as he is concerned are a mixture of talc mineral with various asbestiform minerals and therefore there is a risk of exposure to it. He wants as a very minimum to keep the present restrictions on talc meaning the levels for fibrous talc containing tremolite. Dr. Langer ended up by saying that there is such a thing as fibrous talc which is different than platey talc and different than asbestos. In essence he had no hard facts to present but just proceeded in every manner possible to cast suspicion on talc.

The following speaker was Dr. William Nichol森, also from Mt. Sinai. He again reiterated that asbestos standards should be applied to talc standards. He stated that experience has taught that it takes 30 to 35 years for cancer to appear as a result of asbestos exposure and he does not see any reason for talc to be different. To date he claims that talc data tends to be parallel to the asbestos data for exposure in New York State miners. He emphasizes that the deaths that have been seen to date have been caused by pneumoconiosis, which generally occurs before the onset of respiratory carcinoma. He indicated that he had seen some data that indicated that

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fibrous talc exposure is worse than exposure to chrysotile. He also said that you couldn't say that one form of asbestos was better or worse than any other form and that fibrous talc should be considered in the same camp as asbestos. When questioned he would not define what he meant by fibrous talc, but he did say that he thinks that fibers over 3 microns in diameter are not readily respirable and therefore do little damage. He feels that the present standard of 5 fibers per cubic centimeter by optical microscopy is a bare minimum, his thesis being that for every fiber seen by optical microscopy there are "hundreds" more which would be seen if transmission electron microscopy were used rather than optical microscopy. He stated that on balance the current standard is probably not adequate. As far as the use of fibrous talcs in a consumer area was concerned he felt that they should be labelled with cautionary statements on any consumer goods that can be dispersed into air. He stated that any talc that contains asbestiform minerals or that was a fibrous talc should have the asbestos standards applied to it and that you should use optical microscopy and treat all fibers that you see as if they were asbestos fibers. He also felt that there is technology available at present to meet these standards. Upon closer questioning he agreed that his remarks were limited to New York State tremolitic talcs containing asbestiform minerals. He further agreed that he didn't know anything about other talcs and couldn't say anything about standards for platey talcs.

The last speaker was a Dr. Sykes, representing Ralph Nader Associates. He introduced himself as a chemist and said that he was interested in the safety of cosmetics, that he had no hard data or facts to present but merely wanted to raise "questions". He stated that talc powders are widely used in consumer goods and that his group was concerned that asbestos fibers might be present in them. He quoted both Lewin and Langer on their analyses of commercial products, that a lot of talc products contain 2 to 4% of asbestos with some as high as 20% and that some fell into a borderline area of questionable detection at the 1 to 2% level. He further stated that it was Nader Associates' view that consumer products should be made free of asbestos by some method. He then picked up the theme presented by Pfizer and claimed that electron microscopy was the only definitive way of analyzing talcs and that optical and X-ray methods were not sufficiently sensitive. He said he wanted to raise the issue of what is known about the safety and toxicity of pure talc, and that his group recently has instituted a complete survey of the medical literature to determine this. He said that he felt that the burden of proof was on the people making the consumer products because some doubts had already been raised on the association of talc with the onset of various disease states. He quoted the NIOSH model as indicating this. He also spoke about respiratory cases involving the use of baby powders that had been brought to their attention from the medical literature and also a case where pneumoconiosis had been diagnosed in a patient who was a Q.C. inspector who tested aerosols containing talc. The person had worked one week out

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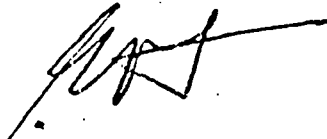
May 18, 1973

of six in such a testing program and had developed pneumoconiosis after several years exposure to such a work situation. He quoted some of the known cases of accidental inhalation of large amounts of talc by children where deaths were known to have occurred. He quoted a "Bernardi" study in reference to this. He ended up on the note that "maybe talc is not totally inert".

Summary:

On overall balance I think the total presentations made were neither as bad as they might have been nor as good as they might have been from our viewpoint. Only once did anyone actually touch on whether the physical form, the fact that fibers per se are the positive cause come up. This was in Langer's presentation where he started to talk about surface chemistry but he never concluded in making his point. There was probably sufficient evidence presented to indicate that talcs do fall into several categories and that pure talcs, meaning platey talcs or talcs not known to be admixed with tremolite or asbestiform minerals, exist and, accordingly, a different standard should be associated with these talcs than with the admixed talcs. It would appear that if the usual decision is made on the cautionary side, then talcs containing tremolite or asbestiform minerals will be pushed into the asbestos or fibrous talc category and the burden of proof will be placed on the manufacturer to show that he has a "pure" or platey talc which should not be in this category. However, it does appear that they will allow the category of a "pure" talc.

I taped the entire 5 plus hours of the symposium. If anyone is interested in listening to all or part of it, please let me know and I will send the tapes.



G. E. Heinze

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Exhibit 148

NOTES ON MEETING WITH PROFESSOR F. POOLEY, CARDIFF, 30.9.92

Present: Prof. Fred Pooley, Dr Alan Gibbs (pathologist). From CTPA: Bernard Wells, John Hopkins, Ian Phillipson. Bernard Miller sent his apologies. Owing to vehicular indisposition, John Hopkins and Ian Phillipson were able to be present only part time.

OVERVIEW The main point which emerged very early in the meeting and which was reinforced many times subsequently was that neither optical nor X-ray diffraction methods are sufficiently sensitive to detect asbestiform fibres of biologically relevant dimensions at the level which the industry now requires. Transmission electron microscopy is the method of choice and should be seriously considered as a supplement to routine screening methods, for instance on first examination of material from a new source and occasionally on samples from current sources.

SPECIFIC POINTS DISCUSSED Similar points which arose during discussion of a number of different issues have been merged in the following summary ie it is not a chronological report of the meeting. ABW had attended a Royal Society of Chemistry meeting the previous day on man-made mineral fibres in which Prof. Pooley and Alan Gibbs were very interested and this prompted a number of discussions relevant to the talc issues, although for clarity and conciseness the MMMP aspects have been omitted from these notes.

A. ANALYTICAL ISSUES

Methods of asbestos fibre detection re HSE, CTPA spec, Dangerous Substances and Cosmetics Directives, and purity of talc re ovarian cancer

Prof. Pooley has not tried to detect fibres in milled talc by optical microscopy. He agreed that since optical microscopy eg by use of the McCrone optical dispersion method is not at a very high magnification, fibres might be missed or not identified.

His thinking on the likely presence of serpentine asbestos (chrysotile) in talc has not changed despite having looked at more lung and mineral samples since the previous meeting. He is not aware of any publication showing the presence of chrysotile in talc although he has (rarely) seen it himself. Detection of chrysotile by XRD is a problem in the presence of chlorite because the peaks overlap, but detection is no problem with TEM. The low mass of a chrysotile fibre means that very high numbers of fibres are present for a low level of contamination, but TEM is necessary to see them.

Prof. Pooley never uses optical methods for looking for contaminants. His reasoning is as follows. The US Federal method for counting asbestos fibres in samples from airborne dust specifies TEM for sufficient sensitivity, therefore it is appropriate to use the same method on mineral samples. As an example, Prof. Pooley found in samples of amosite from houses a 40x ratio of concentration measured by TEM count to that measured by optical microscopy. (This was unpublished contract work for a local authority.)

Long thin fibres are retained in the lungs. -- TEM is the only suitable method for looking for fibres of biologically relevant dimensions in lungs, therefore it is logical to use the same technique for examining mineral products for biologically relevant fibres.

Prof. Pooley is currently looking at tremolite in chrysotile samples. About 50% of the samples are positive but the morphology of the tremolite varies greatly - some of the particles are far too large to be respirable and are therefore biologically irrelevant.

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The CTFA excludes TEM analytical methods on the grounds that they are unsuitable for normal quality control application. Professor Pooley was not convinced that this would now be an acceptable argument, especially in the USA, if the industry wanted to establish the absence of asbestiform contaminants by state of the art methods.

What is clinocllore? This is simply a chlorite, as is the mineral which Dr Toussaint was unable to identify. There are several chlorite minerals which may contaminate talcs, and the presence at a level of 20% by no means indicated addition as an ingredient; levels of 50% or more occur in some Luzenac talcs. Energy dispersive electron microprobe analysis (EDAX) is needed to identify the chemical constituents under high magnification although in the classic paper on the chlorite they were identified by wet chemistry. (Prof. Pooley suggested the following reference for further information which Ian might consider sending to Dr Toussaint: Hey MH 1954 Min Mag 30 277 "A new review of the chlorites" (from Deer, Howie and Zussmann, Rock Forming Minerals, the Sheet Silicates p160). (ABW has ordered a copy of this.)

How to increase the sensitivity of the XRD method (re Cyprus minerals comments and differences between the CTFA and CTFA methods which ABW raised.)

Increasing the counting time from 5 to 30 min. will help with quantitation but not significantly with detection. The differences in the sample preparation method between CTFA and CTFA should not significantly affect the detection limit. Prof. Pooley suggested that the technique most likely to improve the sensitivity significantly is spinning the sample during counting but a spinning goniometer costs about £15k.

The operating conditions should be optimised for the user's machine and not specified in the method. For example, Prof. Pooley's machine is operated at 30kV and 25mA, values very different from those quoted by Cyprus.

It is impossible to be dogmatic about detection limits since not all amphiboles respond in XRD on a mass basis in the same way.

The CTFA specification uses a somewhat different method of sample preparation and analysis compared with the CTFA eg a boric acid backing coat and they do in fact suggest counting over the peak for greater sensitivity. ABW suggested there might be value in comparing analysis of the same samples by the CTFA and CTFA methods; Prof. Pooley agreed, although he would not expect to find a great difference.

Other points raised by Cyprus were on technical issues relating to talc as a personal product ingredient on which Prof. Pooley had no particular views and minor points on mineralogy with which he concurred.

B. BIOLOGICAL ISSUES

NTP study Prof. Pooley and Alan Gibbs had no doubts that the results of the NTP study were attributable to pulmonary overload. ABW mentioned that a similar situation concerning tumour formation in laboratory animals by man-made mineral fibres was going to be investigated by a number of experts in different centres in the USA by means of mathematical modelling. This work was going to take six months, so it seemed unlikely that the talc data had received anything like as thorough an investigation. (See also below re the ovarian cancer issue.)

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Fibre concentrations in lung tissues Diffuse pulmonary fibrosis and lung cancer caused by asbestos fibres are dose dependent and have thresholds of lung fibre concentration. However, on present data the fibre concentrations in the lungs of cases with mesotheliomas and in control lungs without mesothelioma overlap.

Prof. Pooley and Alan Gibbs are currently studying non-occupational exposure to asbestos in a case-control study in the Leeds Armley district and two other districts around the former Roberts' asbestos (crocidolite) factory. Alan Gibbs receives the specimens and is studying the pathology, Professor Pooley is analysing the tissue for minerals. The study is well under way and should be finished in 6-9 months time. If any study is going to show a threshold fibre concentration for mesothelioma development, this is it. Some diffuse fibrosis is often seen in the cohorts where mesotheliomas occur which suggests some relationship with exposure: this does not apply to pulmonary plaques where there is no relationship with mesothelioma development.

Prof. Pooley and Alan Gibbs pointed out that mesotheliomas are not only caused by fibres, as Potts had shown.

Talc and ovarian cancer. Prof. Pooley had nothing new to add on this subject, other than on the point which I made about the evidence from human exposure that asbestos-free talc does not cause tumours in miners and millers heavily exposed. He has completed a study on the fibres in the lungs of Vermont and Luzenac miners and millers. The material in the lungs matches that to which they were exposed, which did not contain amphiboles. This paper is in the press (Human Pathology).

The study confirms that a high lung talc burden is needed for talcosis in primary talc workers. However, the lung samples show that alleged "talc" exposure of secondary talc workers, eg in the rubber industry, is not necessarily just talc, hence epidemiological studies on such workers must be viewed with caution.

Prof. Pooley is currently examining lung tissues from miners exposed to talc in Japanese talc mines. None of those present knew of this source. Do any members of the Talc Subcommittee know of talc mined in Japan?

C. PROF. POOLEY'S CURRENT WORK

The emphasis of the teaching work of the Department has changed from mining to environmental studies. Prof. Pooley is still examining lung samples, mainly for toxic metals especially Cr and Ni.

In a new venture he is trying to see whether "organically" exposed lungs can be characterised by thermal desorption of vapours from lung tissue samples, starting with smokers vs. non-smokers. This is part of a DoH brief for work on the effects of "general atmospheric pollution".
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